AN INVESTIGATION INTO THE EFFECT OF REFLUX RATIO ON HEIGHT OF A TRANSFER UNIT

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OF REFLUX RATIO ON HEIGHT OF A TRANSFER UNIT

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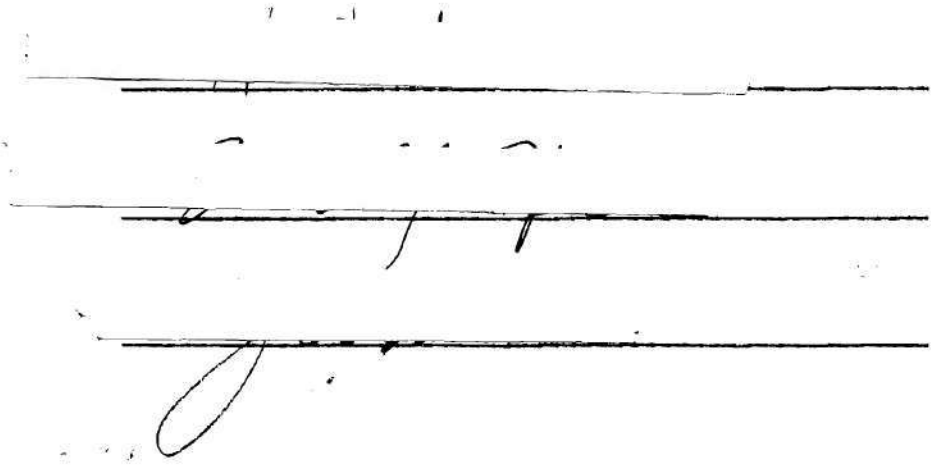

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ABSTRACT

An equilibrium still incorporating a wetted-wall column has been constructed in which the reflux ratio, V/L may be varied from zero to high values. Rectification runs were made with the system carbon tetrachloride-toluene in which V/L varied from 0.331 to 2.97. Vapor velocities were restricted to the transition region between streamline and turbulent flow, with values of ReG ranging from 1600 to 2700.

It was found that the data from these runs could be correlated by using the method of Lewis for evaluating m, the slope of the equilibrium curve, in the theoretical transfer unit equation

$$HTU_{OG} = HTU_G + \frac{mV}{L} HTU_L.$$

Extending Lewis's evaluation of m, there are two distinct values available; one, m', for the HTU_{OG} equation, the other, m", for the equation

$$HTU_{OL} = HTU_L + \frac{L}{m"V} HTU_G.$$

These averages make possible an indirect method of determining HTU_L and HTU_G. Further work will be needed to substantiate the use of m' and m".

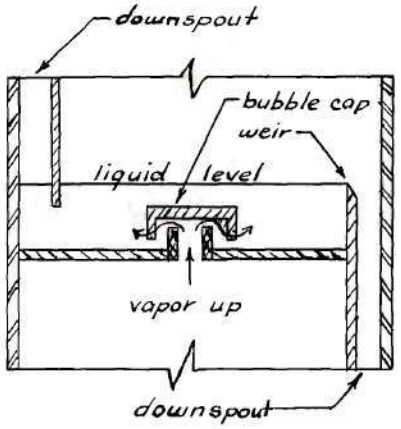
AN INVESTIGATION INTO THE EFFECT OF REFLUX RATIO ON HEIGHT OF A TRANSFER UNIT

INTRODUCTION

"The unit operation which consists of bringing a stream of liquid and a stream of gas countercurrently into direct contact to allow spontaneous interphase transfer of their constituents and of enthalpy is called 'rectification' if the incoming liquid is supplied by condensing part of the gas leaving, or if the incoming gas is produced by reboiling part of the liquid leaving," (5).

The equipment used to bring about countercurrent gas-liquid contact is fairly well standardized, (1, 17, 22). The actual contact takes place in a column or tower. The gas is usually provided by vaporizing in a "boiler" part of the downflow or liquid passing down the column. That part of the downflow which is not vaporized in the boiler is drawn off as "bottoms." The vapor from the boiler passes up the column where it makes intimate contact with liquid passing down the column. After having been thoroughly "scrubbed" with liquid, the vapor leaves the column and is condensed in a condenser. The condensate is then divided, part returning to the top of the column as "wash" liquid or reflux, the remainder drawn off as a product. This product is usually the primary purpose of the rectification. The feed which may be a liquid, a vapor, or a mixture of vapor and liquid, is generally fed to the column at some point between the top and bottom. There are various types of rectification, the type described above being one of the simplest.

This work is concerned primarily with the column used for bringing the gas and liquid into contact. There are two general types of columns - the plate column and the packed column. The principal type of plate column is often referred to as a bubble-cap tower. A simple bubble-cap consists of a short nipple on which a circular cup-shaped cap rests. The vapor passes up through the nipple, reverses direction and flows down through the annular space between the outside of the nipple and the inside of the cap, thence through slits in the sides of the cap and into the liquid as a stream of fine bubbles. The tray, or plate, supporting the bubble-cap, may support many bubble-caps, depending on the tower design. The tower may have as many as fifty trays, depending on the difficulty of the separation involved. The liquid, fed to each tray from the tray above by means of a downspout, flows across the tray, over and around the bubble-caps, to a weir at the top of the downspout feeding the next tray below. The weir height determines the depth of liquid on that tray. A column made up of one or more such trays is generally referred to as a plate column.



The other principal type of column, and the type this work is concerned with, is referred to as a packed column. The interaction between vapor and liquid can be efficiently secured by filling a plain tower with suitable packing material. Much work has been done on the selection of the most economical packing material for many liquid systems. Depending upon the system, packing materials have been recommended ranging from coke lumps and broken beer bottles dumped into the tower, to a large assortment of

specialty packing materials such as Berl saddles, Lessing rings, Stedman packing, and McMahon packing. The choice of packing material is dependent on the economics of the particular case, the corrosiveness of the system, as well as the amount of the gas-liquid interface required. Some means is provided in packed columns for evenly distributing the liquid reflux over the top of the packing to prevent channeling.

In recent years, tests have been made which show that the packed column is very efficient at high reflux ratios. Other potential advantages of the packed column over the plate column are (a) lower pressure drop, (b) lighter weight, (c) shorter column height, and (d) cheaper and simpler construction. Consequently, the industrial use of the packed column has shown some increase in the last few years, and research and development on this type of equipment has increased many times.

The performance of wetted-wall columns is of interest for several reasons - primarily because the data throw light on the mechanism of mass transfer for the systems studied. The interfacial area is fixed and definite and the conditions of turbulence in the gas stream are standard and reproducible. Such data, when available, may be used in the solution of many problems in engineering design.

The addition of packing to a wetted-wall column and the addition of a liquid distributor and a vapor distributor at the top and bottom of the column, respectively, convert the column to a simple packed column. This greatly increases the interfacial area, as well as decreasing the film thicknesses at the interface, but the mechanism of mass transfer remains the same.

Packed columns have been used for countercurrent contacting devices

for many years. Probably the first publication directly describing a packed column, for laboratory use, was that by Hempel (9) in 1881. However, the development of methods of calculation and consequently the use of packed columns for distillation was retarded by a lack of understanding of rectification. These were first expressed in useful mathematical form by Sorel (21) in 1899.

Peters (19) is generally credited with being the first to formulate a mathematical concept for the comparison of the fractionating abilities of different heights of packed columns and the different sizes and types of packings. In the case of packed column distillation, his "Height equivalent to a theoretical plate" (or H.E.T.P.) is the height of packing that will give the same separation as one theoretical plate; that is, a section of packing of a height such that the vapor rising from the top of the section will have the same composition as the vapor in equilibrium with the liquid flowing down from the bottom of that section. In order to calculate the height of column required for any desired separation, if experimental values of H.E.T.P are known, it is only necessary to "step-off" the required number of theoretical plates on a McCabe-Thiele (15) diagram, and multiply this number by the appropriate H.E.T.P.

Peters's concept has been very useful in the design of packed columns. However, it is based on the concept of plates or steps as would occur in a plate column. The enrichment in a packed column actually takes place in an infinite number of differential changes.

The recent introduction of the transfer unit concept by Chilton and Colburn (2, 3) treats the packed column from a fundamental standpoint.

Peters's H.E.T.P. varies sharply with mass velocity, as does the mass

transfer coefficient, K. H.E.T.P. has only one dimension, length, unlike the transfer coefficient. The height of a transfer unit (or HTU) also has only one dimension, length. But, unlike the H.E.T.P., HTU, in many cases, varies only slightly with mass velocity. Hence, approximate values may be easily kept in mind.

The apparent utility of the transfer unit concept is appealing. However, universal adoption has been retarded by the lack of a thorough experimental evaluation. It was the purpose of this work to construct an equilibrium still incorporating a wetted-wall column with which an experimental study of this concept could be instigated.

PREVIOUS WORK

Following Chilton and Colburn's introduction of the transfer unit concept in 1935, the data of Gilliland and Sherwood (8) on the vaporization of pure liquids were recalculated showing the dependence of HTU on the Reynolds number, the Schmidt number, and the column diameter in a wetted-wall column. This equation,

$$\frac{HTU_{OG}}{d_t} = 10.9\ (Re_G)^{0.17}\ (Sc_G)^{0.56}$$

agreed with the equation of Chilton and Colburn,

$$\frac{HTU_{OG}}{d_t} = 10.9\ (Re_G)^{0.2}\ (Sc_G)^{0.67}.$$

The latter equation is not restricted to the vaporization of pure liquids. However, both equations require the Reynolds number of the gas phase to exceed 2100. In both of these equations, Re_G and Sc_G refer to the gas phase and u, the vapor velocity, is taken relative to the column wall.

Johnstone and Pigford (10) published an equation based on data similar to the two above equations but differing in that the Re was calculated with the vapor velocity relative to the moving liquid surface. Johnstone and Pigford also obtained data for the rectification, at total reflux, of three binary mixtures, toluene-ethylene dichloride, ethyl alcohol-water, and acetone-chloroform. Their equation is

$$\frac{HTU_{OG}}{d_t} = 7.63\ (Re_G)^{0.23}\ (Sc_G)^{0.67}.$$

Their values of the Re_G were in excess of 13,200.

Cohen (4) states that the value of HTU_{OG} is proportional to the column diameter and approximately proportional to $Re_G \times Sc_G$ except at low values of the product where $\frac{HTU_{OG}}{d_t}$ approaches a minimum value of 0.48 at $Re_G \times Sc_G = 4.19$ due to the increasing influence of diffusion parallel to the axis of the column. He concludes mathematically that in cases of laminar flow of both phases, the liquid phase resistance is less than 10% of the vapor phase resistance provided the liquid layer thickness is less than $0.015d_t$.

Cohen's equation is

$$\frac{HTU_{OG}}{d_t} = 0.224\frac{(1 + (Re_G\, Sc_G)^2)}{Re_G Sc_G}.$$

Peck and Wagner (16) presented a relationship which provides a means of estimating the fraction of the total resistance provided by the gas phase. The derivation is limited to total reflux operation, and, for lack of data on k_L, assumes k_L and k_G constant throughout the tower. Their own data taken with $Re_G \geq 4100$ on the three systems, methyl alcohol-water, acetone-water, and isoproponal-water, indicated the gas phase resistance to be 94% to 100% of the total resistance to mass transfer in a wetted-wall tower. Data from the literature on wetted-wall towers substituted in their relationship showed 90% to 100% of the total resistance to mass transfer to be in the gas phase.

Suroweic and Furnas (22) conducted tests in a wetted-wall column with an alcohol-water mixture from which they concluded that HTU is a complex function of the properties of the system and hence could be represented only by very complex equations. These equations are:

$$HTU_G = 3.87\alpha \left(\frac{du\rho}{\mu}\right)_G^{0.17} \left(\frac{\mu}{\rho D}\right)_G^{0.56}$$

$$HTU_L = \beta \frac{M}{M_L} \left(G^{4/3} \mu L^{1/3}\right)^{2/3}$$

$$HTU_{OL} = 0.033Z \frac{M}{M_L} \left(\frac{D_L Z \rho_L}{3\Gamma B_L}\right)^{-2/3}$$

$$+ 3.87\alpha \left(\frac{d_t u \rho}{\mu}\right)_G^{0.17} \left(\frac{\mu}{\rho D}\right)_G^{0.56} \left(\frac{L}{mV}\right)$$

$$HTU_{OG} = 3.87\alpha \left(\frac{d_t u \rho}{\mu}\right)_G^{0.17} \left(\frac{\mu}{\rho D}\right)_G^{0.56}$$

$$+ 0.033Z \frac{M}{M_L} \left(\frac{D_L Z \rho_L}{3 B_L \Gamma}\right)^{-2/3} \frac{mV}{L}$$

where $B_L = \left(\frac{3\mu_L \Gamma}{\rho_L^2 g}\right)^{1/3}$.

Furnas and Taylor (7) suggested averaging the slope of the equilibrium curve over the column in a manner similar to that used in averaging the values of heat capacities of gases. Thus, they defined

$$m_{av} = \frac{\left[\int_0^{y_2^*} m dy\right] - \left[\int_0^{y_1^*} m dy\right]}{y_2^* - y_1^*}.$$

This value of m has been used in the fundamental HTU equation of Chilton and Colburn with the same success. However, m_{av} has little in the way of theoretical support for this usage.

Suroweic and Furnas used a value of m, designated as $\bar{m}$, which was

defined

$$\overline{m} = \frac{L}{V} \cdot \frac{HTU_{OG}}{HTU_{OL}} .$$

As will be shown later, this will be true if, and only if, the liquid-vapor equilibrium curve is known or may be assumed to be a straight line.

Ducan, Koffolt and Withrow (6) conducted tests in a packed column five inches in diameter and packed with $\frac{1}{2}$ inch Raschig rings to a height of nine feet. The tests were conducted over a period of five years and used five systems to study thoroughly the effect of m, the slope of the equilibrium curve. The binary systems studied were:

1. Carbon Tetrachloride-Benzene
2. Trichloroethylene-Toluene
3. Methanol-Water
4. Acetone-Water
5. Ethanol-Water

Their investigation brought out the following points:

"1. The performance of a packed column was found to be affected by the vapor-liquid ratio, $G/\overline{L}$, the average slope of the vapor-liquid equilibrium diagram, m_{av}, and the rate of throughput.

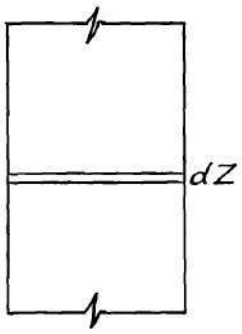
"2. Plotting HTU_{OG} versus m_{av} with $G/\overline{L}$ as a parameter gave a satisfactory, practical and simplified correlation of the results."

Ducan, Koffolt and Withrow used the same method of averaging m, the slope of the equilibrium curve, as that used by Furnas and Taylor (7).

THEORY

Considering only binary mixtures, it is assumed that owing to the diffusion of the more volatile component in the vapor, the concentration of this component in the vapor increases continuously as the vapor passes upward, rather than in steps as in a plate column.

Consider a section of a packed column of height Z. Over a differential height, dZ, the partial pressure of the low boiling component in the vapor is increased by a differential amount, dp, and since in distillation, the total moles of vapor passing a given point remains essentially constant, the differential rate of increase of moles of low boiling component in the vapor is given by



$$dW = \frac{GSdp}{\pi M_m} \text{ - - -

$$\frac{dp}{\Delta p} = \frac{Ka\,\pi M_m\,dZ}{G} \quad \text{- - - - - - - - - - - - - - - - (3)}$$

To obtain the height of column required for any given separation, integrate Equation 3,

$$\int_{p_1}^{p_2} \frac{dp}{\Delta p} = \frac{Ka\,\pi\,MmZ}{G} \quad \text{- - - - - - - - - - - - - - - - (4)}$$

and solve for Z,

$$Z = \frac{G}{Ka\,\pi\,M_m} \int_{p_1}^{p_2} \frac{dp}{\Delta p} \quad \text{- - - - - - -(5)}$$

Thus, if the value of Ka could be predicted, the height of a column to produce any desired separation may be determined.

Consider, however, the quantity:

$$\int_{p_1}^{p_2} \frac{dp}{\Delta p}.$$

This quantity may be thought of as a measure of the difficulty of any desired separation - in a manner similar to considering the required number of theoretical plates as a measure of the difficulty of a separation to be carried out in a plate tower. This similarity led Chilton and Colburn (2) to refer to the above integral as, "The number of transfer units." Thus the number of transfer units is

$$N_{OG} = \int_{p_1}^{p_2} \frac{dp}{\Delta p} = \int_{y_1}^{y_2} \frac{dy}{\Delta y} \text{ - - - - - - - - (6)}$$

if the driving forces are expressed in terms of mole fractions instead of partial pressures.

Thus, without considering the total moles being transferred, any problem may be expressed as the required number of transfer units. Hence, if experimental data are available as to the value of the height per transfer unit (HTU), the determination of packed column height merely involves the multiplication

$$N_{OG} \times (HTU)_{OG} = Z \text{ - - - - - - - - - - - - - - (7)}$$

As a matter of interest, the relationship between HTU and K is seen from Equations 5 and 6 to be:

$$HTU_{OG} = \frac{Z}{N} = \frac{V'}{Ka} = \frac{G}{Ka \pi M_m} \text{ - - - - - - - - (8)}$$

and

$$\frac{dZ}{HTU_{OG}} = N_{OG} = \frac{dy}{\Delta y} \text{ - - - - - - - - - - - - - - (9)}$$

The HTU concept is similar in some respects to Peters's (19) H.E.T.P.

Determination of the Number of Transfer Units

In rectification, a relationship can be derived between the vapor composition y and the liquid composition x leaving any cross-section above the liquor feed level as:

$$y = \frac{Lx}{V} + (1 - \frac{L}{V})x_p, \text{- - - - - - - - - - - - - - - - - (10)}$$

and below the feed level:

$$y = \frac{Lx}{V} - (\frac{L}{V} - 1)x_w. \text{- - - - - - - - - - - - - - - - - (11)}$$

These equations, known as the equations of the operating lines, are determined solely from the conditions of operation. The reflux ratio, $(\frac{L}{V})$, can be taken as constant on the assumption that the components of the mixture have approximately equal latent heats, negligible heat of mixing and that the temperatures change through the column and the heat losses from the column can be neglected.

These equations with the liquid-vapor equilibrium curve permit the solution of Equation 6 for the general case. This involves choosing a number of values of x between x_1 and x_2, calculating the corresponding value of y from Equation 10 or 11 and at the same value of y, obtaining values of y* from the equilibrium curve.

A plot is then made of $\frac{1}{y^* - y}$ vs y and graphically integrated between y_1 (composition of vapor entering the column - in equilibrium with x_w) and y_2 (composition of vapor leaving the column - product composition) to obtain N = number of transfer units.

Relation to Overall HTU to Single Film Values of HTU (3)

Consider a point in the column where x = mole fraction of solute in liquid, and y = mole fraction of solute in vapor.

Just as the HTU_{OG} may be defined as in Equation 9,

$$\frac{dZ}{HTU_{OG}} = \frac{dy}{y^* - y}, \quad \text{----------(9)}$$

the other values of HTU may be defined in a similar manner:

$$\frac{dZ}{HTU_{OL}} = \frac{dx}{x - x^*} \quad \text{----------(12)}$$

$$\frac{dZ}{HTU_{G}} = \frac{dy}{y_i - y} \quad \text{----------(13)}$$

$$\frac{dZ}{HTU_{L}} = \frac{dx}{x - x_i}. \quad \text{----------(14)}$$

Solution may be made for HTU_{OG} in terms of the single film values of HTU in the following manner:

Eliminate $\frac{dZ}{dy}$ from Equations 9 and 13,

$$HTU_{OG} = HTU_{G} \frac{(y^* - y)}{(y_i - y)}. \quad \text{----------(15)}$$

Now $$\frac{y^* - y}{y_i - y} = \frac{y^* - y_i + y_i - y}{y_i - y}$$

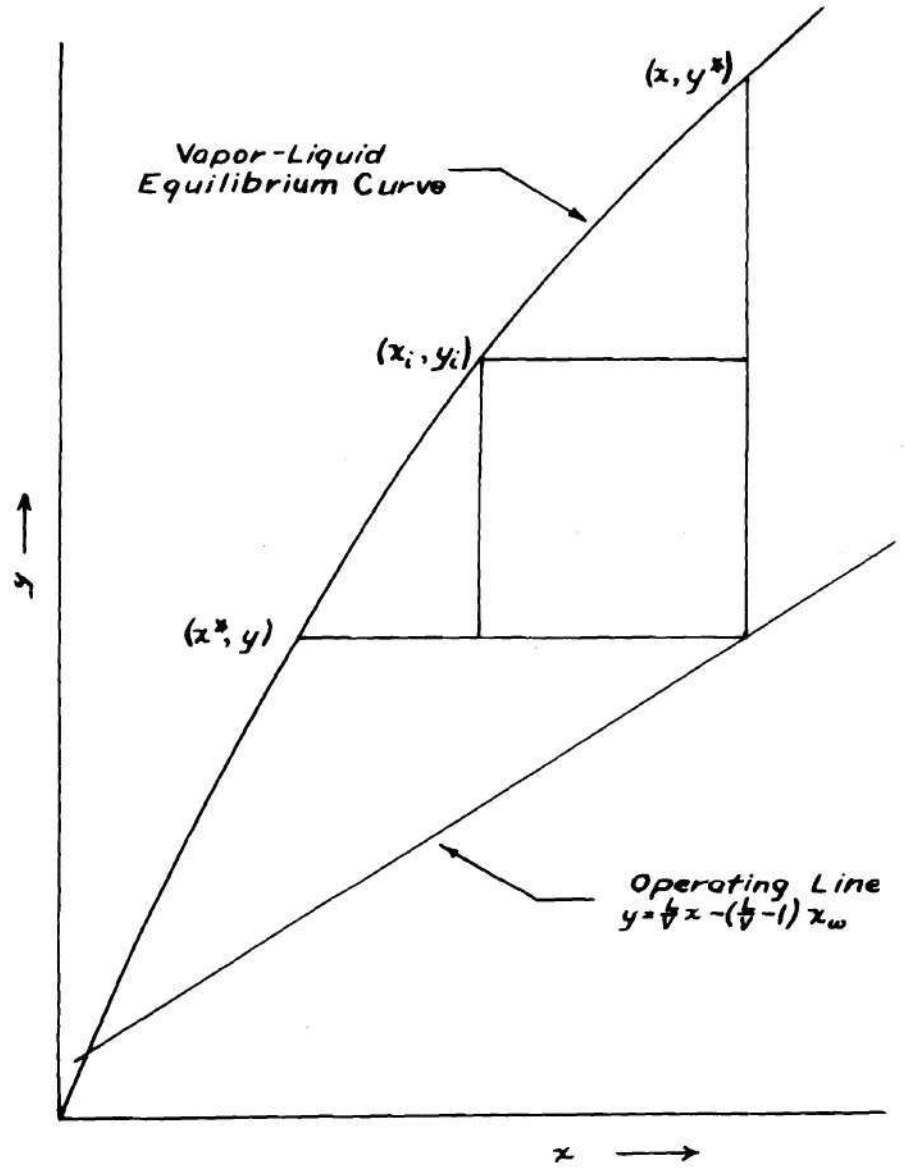
or $$\frac{y^* - y}{y_i - y} = 1 + \frac{y^* - y_i}{y_i - y}. \quad \text{----------(16)}$$

If we consider the equilibrium curve a straight line of slope m over the small range $y^* - y_i$, then:

$$y^* - y_i = m_L (x - x_i). \quad \text{----------(17)}$$

By substituting Equation 17 in Equation 16,

FIGURE 1



Graphical Relationship
Between y, y_i, and y^*

J.F. Kinney

$$\frac{y^* - y}{y_i - y} = 1 + \frac{m_L (x - x_i)}{y_i - y} \quad \text{- - - - - - - - - - - - - - (18)}$$

and Equation 18 in Equation 15,

$$HTU_{OG} = HTU_G + HTU_G \frac{m_L (x - x_i)}{y_i - y} \quad \text{- - - - - - - - (19)}$$

Solving for $\frac{x - x_i}{y_i - y}$ from Equation 13 and Equation 14,

$$\frac{x - x_i}{y_i - y} = \frac{HTU_L \, dx}{HTU_G \, dy} \quad \text{- - - - - - - - - - - - - - - - - - (20)}$$

Writing a mole balance over a differential section of the tower,

$$V dy = L dx$$

or

$$\frac{dx}{dy} = \frac{V}{L} \quad \text{- (21)}$$

Substituting Equation 21 in Equation 20,

$$\frac{x - x_i}{y_i - y} = \frac{HTU_L \, V}{HTU_G \, L} \quad \text{- - - - - - - - - - - - - - - - (22)}$$

and then Equation 22 in Equation 19,

$$HTU_{OG} = HTU_G + \frac{m_L V}{L} HTU_L \quad \text{- - - - - - - - - - - - - - (23)}$$

In a similar manner, HTU_{OL} may be derived as

$$HTU_{OL} = HTU_L + \frac{L}{m_G V} HTU_G \quad \text{- - - - - - - - - - - - - - - (24)}$$

The significance of the term $\frac{mV}{L}$ may be seen more clearly when written $\frac{m}{L/V} = \frac{\text{slope of equilibrium curve}}{\text{slope of operating line}}$.

It may be seen that if m_G does not differ greatly from m_L, solving Equation 23 and Equation 24 simultaneously will produce an average figure for the two, m_G and m_L. Hence, by substituting the value of HTU_G from Equation 23 in Equation 24, and assuming $m_G = m_L$

$$\frac{HTU_{OL}}{HTU_{OG}} = \frac{L}{\overline{m}V}$$

or
$$\overline{m} = \frac{HTU_{OG}}{HTU_{OL}} \times \frac{L}{V}. \quad \text{- - - - - - - - - - - - - - - - - - (25)}$$

If the equilibrium curve is known, or may be assumed to be a straight line, m may be calculated as $\overline{m}$.

However, it should be noted that while

$$m_L = \frac{y^* - y_i}{x - x_i}, \quad \text{- - - - - - - - - - - - - - - - - - (17)}$$

$$m_G = \frac{y_i - y}{x_i - x^*}. \quad \text{- - - - - - - - - - - - - - - - - - (26)}$$

Hence, m_L is the slope of the chord corresponding to the resistance of the vapor phase to diffusion, and m_G is the slope of the chord corresponding to the resistance of the liquid phase. The two slopes are generally quite different. Thus, for the usual case, the result of the substitution indicated in Equation 25 would be:

$$HTU_{OL} = HTU_L \left(1 - \frac{m_L}{m_G}\right) + \frac{L}{m_G V} HTU_{OG} \quad \text{- - - - - - - - (27)}$$

Similarly, $$HTU_{OG} = HTU_G \left(1 - \frac{m_L}{m_G}\right) + \frac{m_L V}{L} HTU_{OL} \quad \text{- - - - - - - - (28)}$$

It will be seen from Equation 8 that HTU_{OG} has a definite relationship to K_Ga. Since K_Ga varies directly as $u^{0.8}$, HTU_{OG} should be substantially independent of gas velocity. Similarly, $HTU_G = V'/k_Ga$, and since k_G varies as $u^{0.8}$, HTU_G may be considered relatively constant with respect to gas velocity. In a like manner, $HTU_L = L'/k_La$. Now $k_L = \psi L^n$, where ψ and n are dependent upon the system considered, and n frequently approaches the value of one. Hence, HTU_L may be considered relatively independent of the liquid flow rate.

Now if HTU_G and HTU_L may be considered constant, according to Equation 23 a plot of HTU_{OG} versus mV/L would produce a straight line. The intercept of this straight line with the HTU axis would be equal to the value of HTU_G, while the slope should produce the value of HTU_L.

It should be noted, however, that a plot of Equation 23 will not produce a perfectly straight line. $HTU_G = V'/k_Ga$ which, since k_Ga is proportional to $u^{0.8}$, means that HTU_G is a function of $(V')^{0.2}$. HTU_G is also known to be affected by the gas viscosity. Similarly HTU_L is slightly affected by the flow rate as well as the viscosity.

Another method for obtaining an average value of m (see pages 8, 9) has been suggested by Lewis (12). Multiplying Equation 23 by Equation 9,

$$Z = HTU_G \int \frac{dy}{y^* - y} + \frac{V}{L} HTU_L \int \frac{m_L dy}{y^* - y} \ .$$

m' may be defined by the relation

$$Z = HTU_G \int \frac{dy}{y^* - y} + \frac{m'V}{L} HTU_L \int \frac{dy}{y^* - y} .$$

Solving for m'

$$m' = \frac{\int \frac{m_L dy}{y^* - y}}{\int \frac{dy}{y^* - y}} . \qquad \text{-----------(29)}$$

Consideration of m' will show it weighs m most heavily in the region where the operating line is closest to the equilibrium curve, hence the region which accounts for the greater part of the height of the tower.

It should be noted that m' corresponds to m_L in the previous equations. A similar derivation developes a corresponding evaluation for the average m_G -

$$m'' = \frac{\int \frac{dx}{x - x^*}}{\int \frac{dx}{m(x - x^*)}} .$$

If further work substantiates the use of m' and m" in averaging m, Equations 27 and 28 should provide a means of evaluating the individual HTU's. And the use of the individual film values of HTU should prove more satisfactory in correlating column performance data than the use of the overall HTU's.

APPARATUS

The primary requirement for the column was the wetted-wall. A liquid distributor had to be used that would evenly distribute the reflux over the circumference of the column. The column should be adiabatic and some means had to be devised in order to approach adiabatic operation. In order to study the variables under stripping as well as exhausting conditions, some means had to be devised to vary the reflux ratio, L/V, from zero to very high values - well in excess of the total reflux value of L/V = 1 (Figure 2). The column material had to be of such a nature that a wide variety of systems could be used without danger of having corrosion effects enter into the problem.

Column

The column proper was fabricated by Ace Glass Company.

The reflux inlet to the reservoir is located at the base of the reservoir and 1/2 inch to 3/4 inch below the overflow lip (Figure 3). This submerged inlet, at the flow rates involved, produces an even overflow of reflux over the lip. So long as the reflux flow rate exceeds a certain critical flow, the column wall will be evenly wetted over its entire length.

The reflux outlet is located at the base of a collector well in the base of the column (Figure 4). The vapor inlet extends up through this well above the reflux outlet and hence prevents any of the liquid flowing down into the boiler through the vapor inlet.

Four holes, approximately 3/32 inch in diameter, were blown approximately 13-1/2 inches apart along one side of the column for thermocouple entrances. These couples, with couples at the top and bottom were designed to measure conditions in the column.

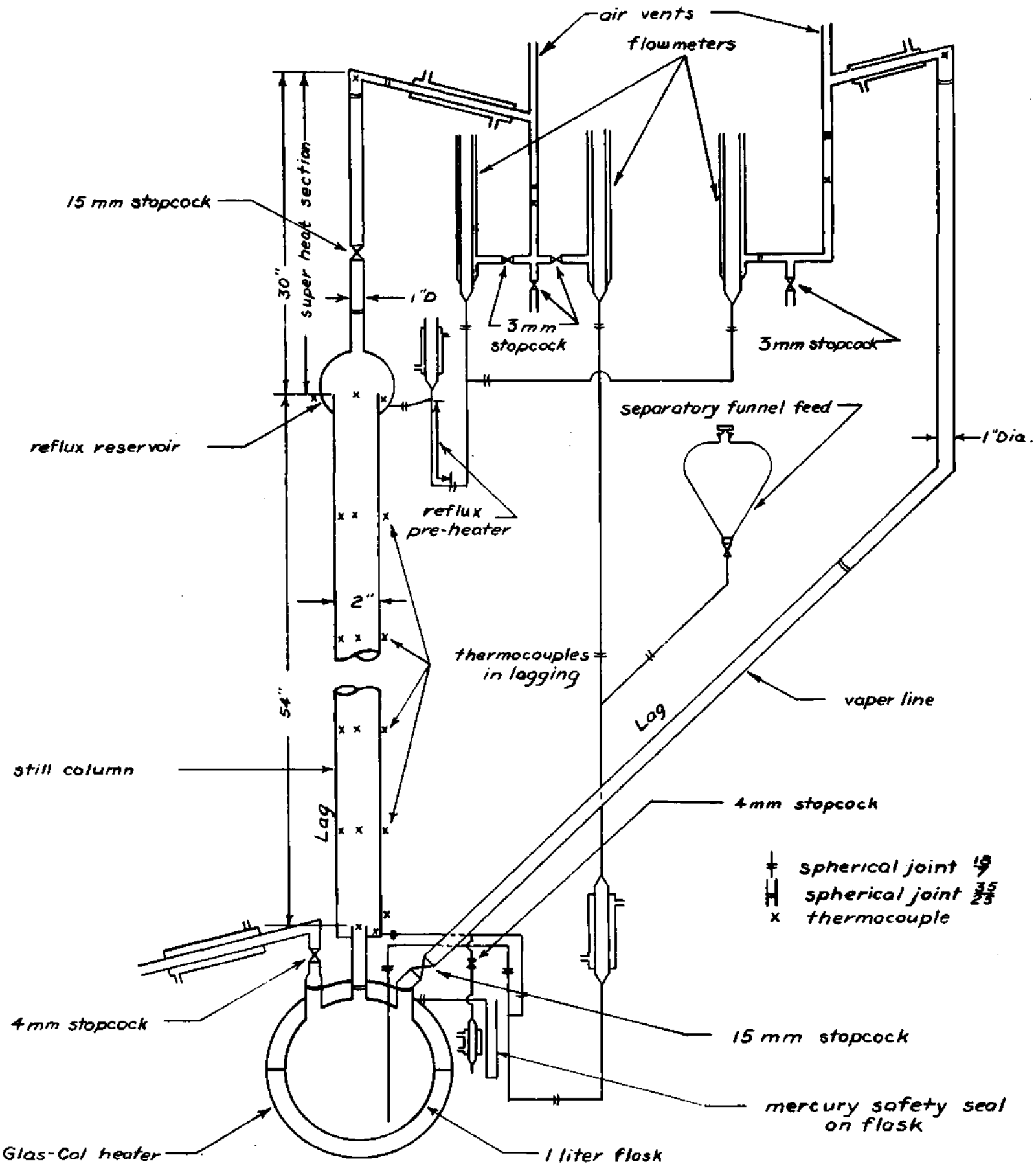
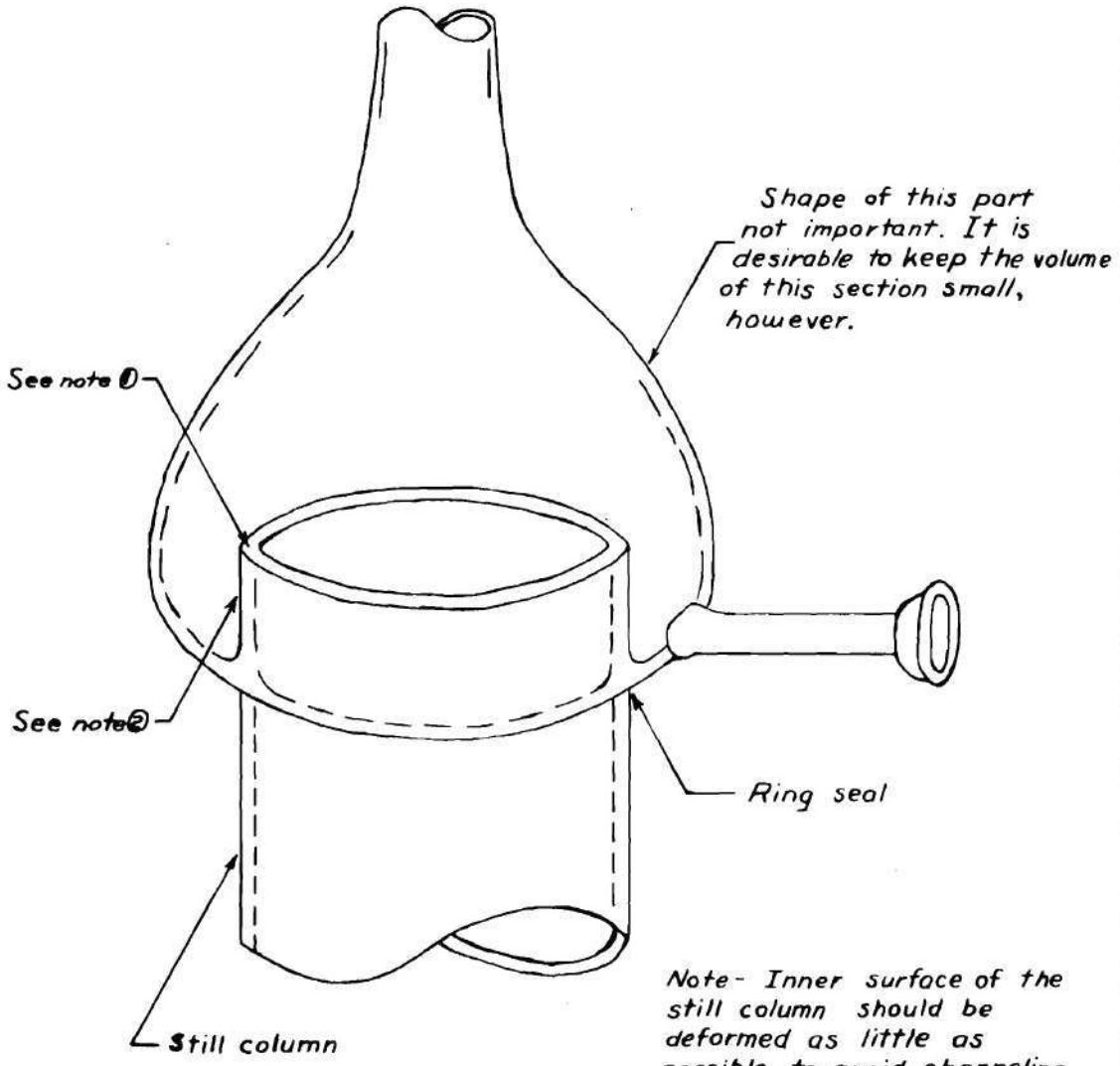


FIGURE 2

DIAGRAM OF APPARATUS

Note ① Top of column to be accurately ground perpendiculor to ℄ of column.

Note ② Enclosed section of column must extend at least ½", preferably ¾", beyond ring seal.



Note - Inner surface of the still column should be deformed as little as possible to avoid channeling of the reflux.

FIGURE 3

DETAILS OF REFLUX RESERVOIR CONSTRUCTION

Mat'l. Pyrex Glass

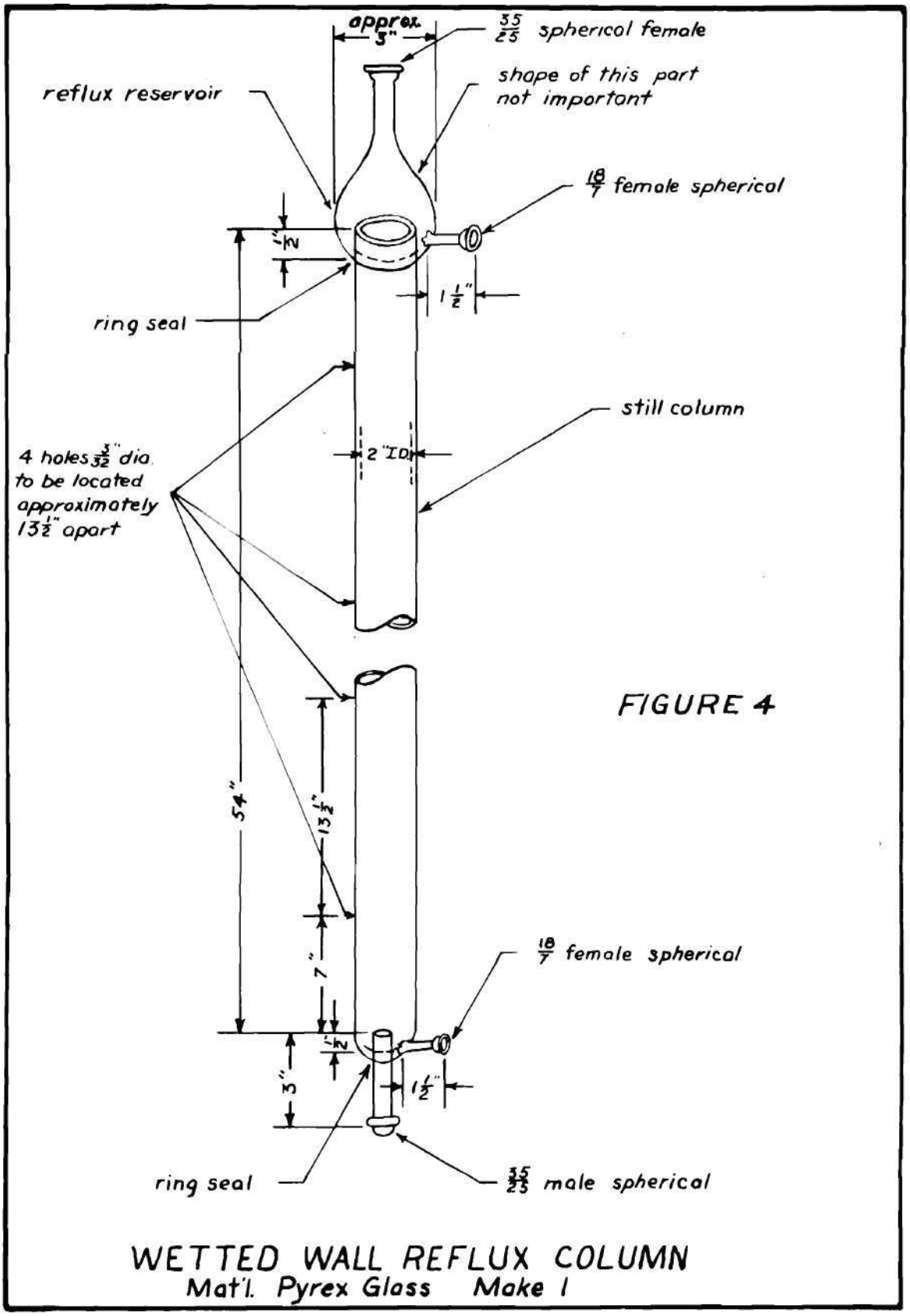


FIGURE 4

WETTED WALL REFLUX COLUMN
Mat'l. Pyrex Glass Make 1

All tubing connections were provided with spherical joints of the appropriate size to facilitate installation and maintenance.

The thermocouples extending into the column were inserted and then held in place with Saureisen - a liquid porcelain cement. The column was then wrapped with 1/8 inch asbestos sheet. Thermocouples in the lagging were installed - held in place by cotton strings - and a second layer of 1/8 inch asbestos sheet placed on the column. The heating wire was then wrapped around the column - consisting of three 600-watt nichrome heating elements, each stretched to about 17 feet, and connected in series. This heating element extended only over the wetted-wall portion of the column.

The column was then wrapped with 3/16 inch asbestos rope and finished with a 1/2 inch layer of magnesia (Figures 26 and 27).

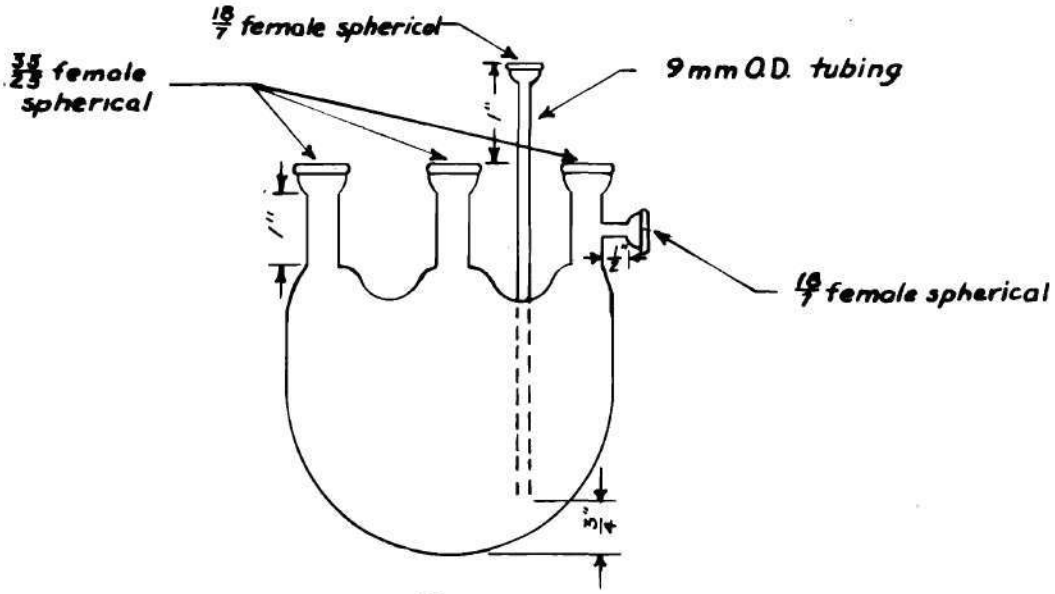
Boiler

The boiler was also fabricated on order by Ace Glass Company. It consists of a one liter, three-necked flask fitted with three 35/25 mm female spherical joints and two 18/7 mm female spherical joints as shown in Figure 5.

A GlasCol, hemisphere type, heating mantle which produces up to 600 watts provides the heat. A similar mantle was molded, using magnesia insulation, to fit over the top of the flask to reduce heat losses from this section (Figure 28). The GlasCol mantle support was hooked to the steel framework, with springs which kept the joints between the column and flask under a slight pressure.

Flowmeters

The flowmeters are of the orifice head type - also fabricated by Ace Glass Company. The orifice type meter was selected to provide the least



Use an ACE #3895 type distilling flask; replace ⅄ joints with spherical joints. 1 liter capacity.

FIGURE 5

BOILING FLASK

Mat'l. - Pyrex Glass

Make 1

change of calibration with change in density and viscosity. The construction of these meters is shown in Figure 6.

Vapor Line

The vapor line provides the extra reflux for the values of L/V greater than one. The vapor line is made up of 1 inch glass tubing, and is provided with several 35/25 mm spherical joints to facilitate assembly and disassembly. The tubing was first wrapped with 1/8 inch asbestos sheet. Then two 600-watt nichrome heating elements, each stretched to about 17 feet in length, were wrapped around the asbestos sheet, one over the top half and the other on the bottom half of the vapor line. Then one layer of 3/16 inch asbestos rope was placed on the vapor lines.

Feed

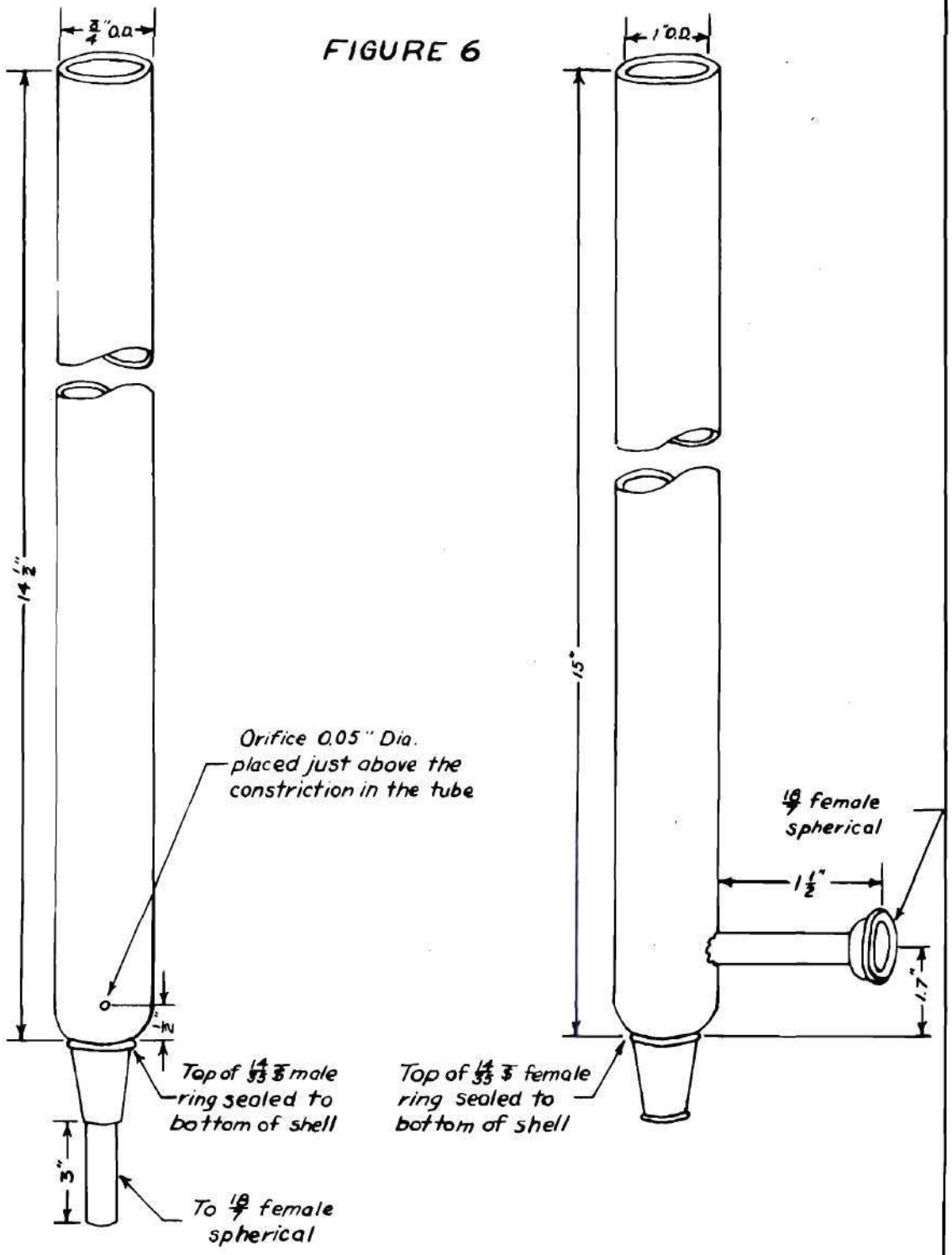
The feed solution may be fed to the pot through a separatory funnel fitted with an 18/7 mm male spherical joint on the draw-off leg.

Superheat Section

The line feeding the vapor from the top of the column to the condenser is 1 inch glass tubing, fitted with a 15 mm stopcock to control relative flow through the column and the vapor line. This section was superheated to prevent any condensation on the wall of the tubing with the resultant fractionation.

This section was fabricated in several sections to provide ease of assembly and disassembly. The superheat line was wrapped with 1/8 inch asbestos sheet and then a 600-watt nichrome heating element was cut in sections proportional to the length of each section of tubing and wrapped around the asbestos covered tubing. The sections were then wrapped with 3/16 inch asbestos rope.

FIGURE 6



FLOWMETERS

Mat'l. - Pyrex Glass Make 3

Reflux Preheater

The line feeding the overheads from both the vapor line as well as the column to the top of the column was wound with a nichrome wire heating element to provide the column with hot as well as cold reflux.

The heated section of the tubing was fabricated from a conventional laboratory reflux condenser of the type used in organic laboratories. The condenser was inverted with the prongs pointed upward to provide mixing of the reflux and was fitted at the bottom with an 18/9 mm female spherical joint. (See Figure 7). The top, in addition to having the feed leg to the top of the column had a small condenser mounted vertically above the preheater. This condenser was necessary, since in many of the runs, the reflux was run in at the boiling point and any loss would necessarily change the operation of the column from the conditions of equilibrium. If the top of the preheater had been sealed off - that is without a condenser, the feed, entering at the boiling point might be a mixture of gas and liquid producing surges through the 7 mm tubing entrace to the column and thus disturbing the smoothness of the wetted-wall surface. The feed leg from the preheater was tilted down at an angle of about 20 degrees from the horizontal to permit any gas bubbles to return to the higher liquid surface under the condenser.

Framework

The support for the column was made up of steel 1-1/2 inch equal angle sections. A box-type frame was constructed to hold the column with leveling screws near the top of the column. The column was supported at the base of the frame on a wooden block carved out to match the curvature of the lower part of the asbestos and magnesia covered column.

This framework, bolted both to the floor and to the ceiling, gave the

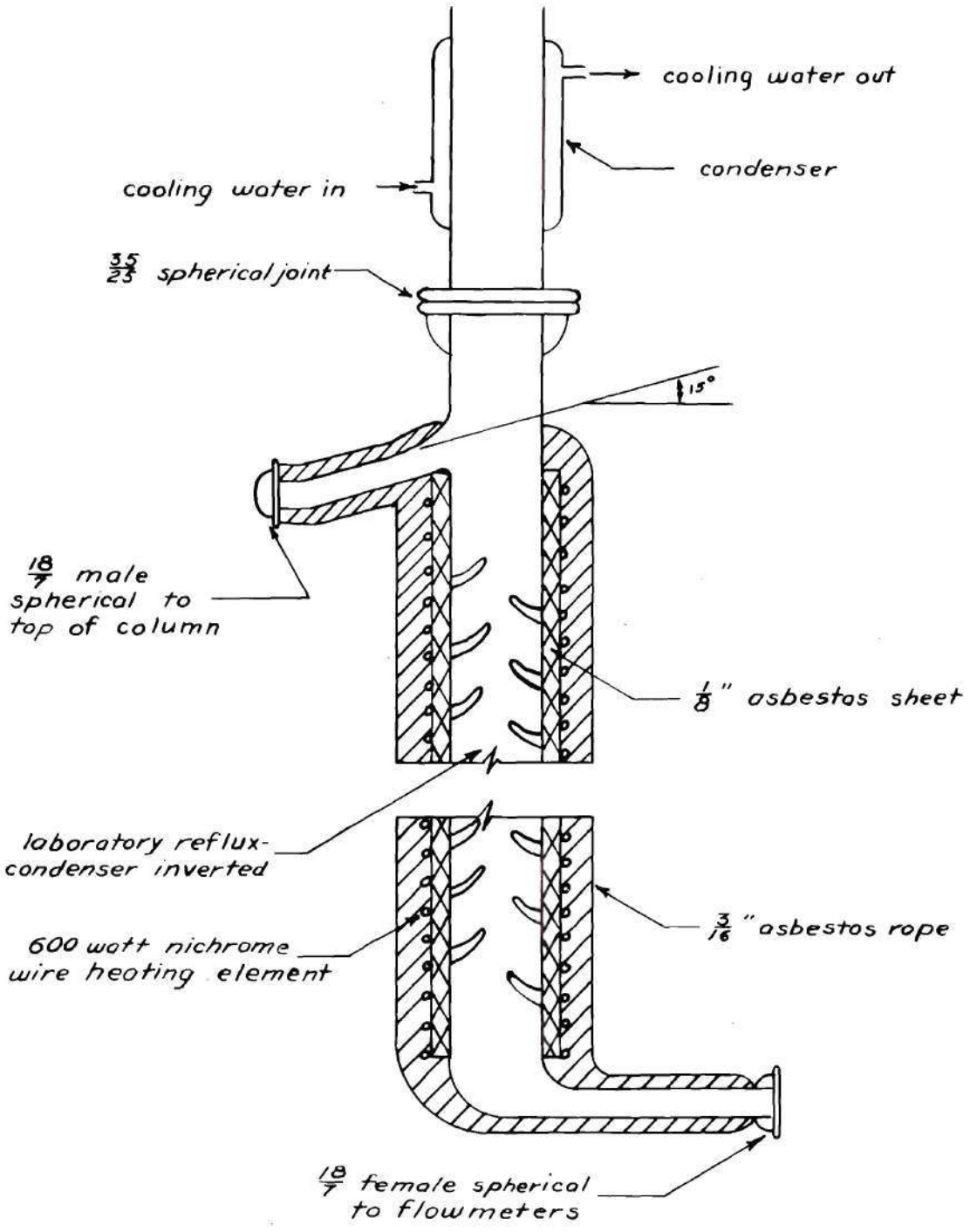


FIGURE 7

REFLUX PRE-HEATER

Mat'l. — Pyrex Glass

Make 1

support needed for the angle-iron and strap-iron cross pieces which supported the 1/4 inch steel-rod uprights which in turn supported the auxiliary glass tubing, flowmeters, etc., in the setup.

Auxiliary Equipment

The thermocouples, twenty-two in number, made up of #24 copper and constantan wires, were connected through switches to a Leeds and Northrup type K potentiometer and thence to a cold junction set in cracked ice in a Dewar flask. Copper leads were used in this hookup.

During the latter phases of the work, a General Electric automatic potentiometer and milliammeter were used to make the thermocouple readings. The use of this potentiometer in place of the type K potentiometer cut the time required for a set of readings from twenty minutes to approximately five minutes.

The heating coils as well as the GlasCol heating mantle were controlled with seven Variacs mounted on a common switchboard. (See Figure 29). Each Variac was connected through a suitable switching arrangement to a common ammeter and a common voltmeter. Thus the current drain of each heating coil could be adjusted and held constant during the run. A constant voltage transformer of the saturable-core type controlled the power input to the switchboard. (See Appendix III).

A Bausch and Lomb precision type refractometer No. BK-574 using a sodium vapor lamp was used in making the analysis. The temperature of the samples was maintained constant during the readings by utilization of a constant temperature water bath.

PRELIMINARY WORK

The preliminary experimental work may be divided into four parts: (1) the selection of a system to use in the tests; (2) calibration of the instruments needed; (3) performing preliminary runs to check operation of the equipment to obtain satisfactory performance; (4) conducting further preliminary runs to establish the proper experimental procedure for the column operation.

Selection of a System

The benzene-toluene system was preferred because of the abundance of data on the system, both physical data as well as operating data. However, probably the only accurate method of obtaining an analysis on the system is by use of the boiling point method using an appropriate boiling-point apparatus. The absence of any departmental equipment of this type eliminated this method, and hence this system.

The toluene-carbon tetrachloride system was promising because of the ease of analysis - either by specific gravity or by refractive index. Also physical data were obtainable in the literature.

The system had a negligible heat of mixing, and though the latent heats showed some variation, this was not deemed serious. These chemicals were readily available, and consequently, this system was chosen.

Calibration of Instruments Needed

a. Flow Rates. The flowmeters were calibrated under actual operating conditions. The boiler was filled with a 50-50 (by volume) mixture of toluene and carbon tetrachloride. Vapors from this solution passed up the column, were condensed in the condenser and were then run through one of the flowmeters. The flow not collected was returned to the boiler. For an interval timed by

a stop-watch, the efflux from the flowmeter was collected in a graduated cylinder. The condensate temperature was checked before and after each collection as read by the thermocouple in the liquid line.

Each flowmeter was calibrated in turn. The condensate temperatures were varied over a twenty degree range and found to cause little variance in the volumetric rate of flow. Of necessity, the composition of the condensate changed somewhat during these runs and provided a check on the variation of flow rate with composition.

These flowmeter calibrations were checked twice during the series of runs.

b. Temperatures. The copper-constantan thermocouples were checked before installation with the boiling points of benzene, water, and toluene, and the melting point of ice. Any couple not producing constant electromotive force of the correct magnitude was replaced at this time. Extra thermocouples were made up and calibrated above the number required in the apparatus. Any couple failing to give constant readings at any time after installation was replaced with another calibrated thermocouple. This precaution paid off many times, since, although it was not forseen, a definite corrosion of the thermocouples was evidenced, and many corroded apart.

The cold junction was maintained at 32^{F} by placing it in a Dewar flask filled with cracked ice. The electromotive force produced by the thermocouple was read by use of a Leeds and Northrup type K potentiometer. The zero setting of the potentiometer was checked prior to each set of readings - both in the calibration of the couples as well as during each run - against a standard cell.

c. Analysis. A Bausch and Lomb precision type refractometer was

used for analyzing the samples taken from the column. The instrument was calibrated by setting the scale at the proper reading, when the correct diffraction pattern had been obtained, for the standard glass specimen supplied with the instrument.

A series of samples were made up gravimetrically ranging from pure toluene to pure carbon tetrachloride. The reading of the instrument was read for each of these samples. A curve was drawn plotting the scale reading of the refractometer versus the mole fraction carbon tetrachloride. Seven points were taken initially. These points were checked with three additional points taken between the seven initial points.

Preliminary Runs

The first run, by its excessively high value of HTU, indicated the possibility of channeling. When the column alignment was checked with a plumb, the column was considerably out of line with the vertical. After aligning, the HTU dropped considerably.

It was decided that in order to check for channeling during the run, three square blocks should be cut out of the insulation at the base of the column. By catching the reflection of a movable source of light from the reflux film on the column wall, the relative evenness of the film flow could be determined. It subsequently was discovered that the floor, on which the frame was mounted was settling, apparently, since the column had to be aligned practically every day.

The first runs also indicated clearly that with the preheater as originally installed, which consisted of an L-shaped piece of 7 mm glass tubing, heated with a nichrome wire heating element and wrapped with asbestos rope, the reflux temperature had to be maintained considerably

below the boiling point of the reflux. When approaching the boiling point, vapors formed in the preheater which, in passing through the 7 mm entrance to the column reservoir, produced surges, disturbing the evenness of the film. In some instances, channeling was detected.

Thus, the preheater was redesigned as shown in Figure 7. This change permitted a much closer approach to the boiling point of the reflux.

At this time, a means of sampling the liquid film out from the bottom of the column was installed. This enabled heat and material balances to be made on the tower to check each run.

Runs Establishing Procedure of Operation

It was found that the use of stopcocks to adjust flowrates is a rather crude method. Consequently, the L/V desired was roughly adjusted and then the run made to equilibrium with further adjustment. Continual manipulation of the stopcocks resulted in a run which would take eight hours or more to obtain equilibrium. By only roughly adjusting the L/V, equilibrium was approached in four to five hours.

Consequently, the reflux ratio was set approximately at the beginning of the run. The voltage was adjusted on the base heater and approximately adjusted on the column heaters, the preheater and the vapor line, if used. If the readings of the thermocouples indicated that any one of the heaters was drawing too little or too much current in relation to the other heaters, this was adjusted during the first two or three hours.

The preheater was adjusted for the majority of the runs to provide hot reflux. It was found that, by adjusting the preheater Variac to provide just enough current through the coil to cause a slight amount of condensation of the vapors in the condenser over the preheater, the reflux would enter only

a few degrees below its boiling point.

When three consecutive sets of readings indicated no appreciable change, that is, no change for a period of one hour, it was assumed "equilibrium" had been attained. The flowmeters were probably the most sensitive to any change.

At this time, the system was sampled. The samples consisted of about three cubic centimeters of each stream, collected in liquid form. The streams were sampled in the following order: the sample of the overhead product was taken first, the vapor line, when used, was sampled secondly; the vapor entering the column was next sampled and then the liquid film from the bottom of the column was sampled.

TABLE I

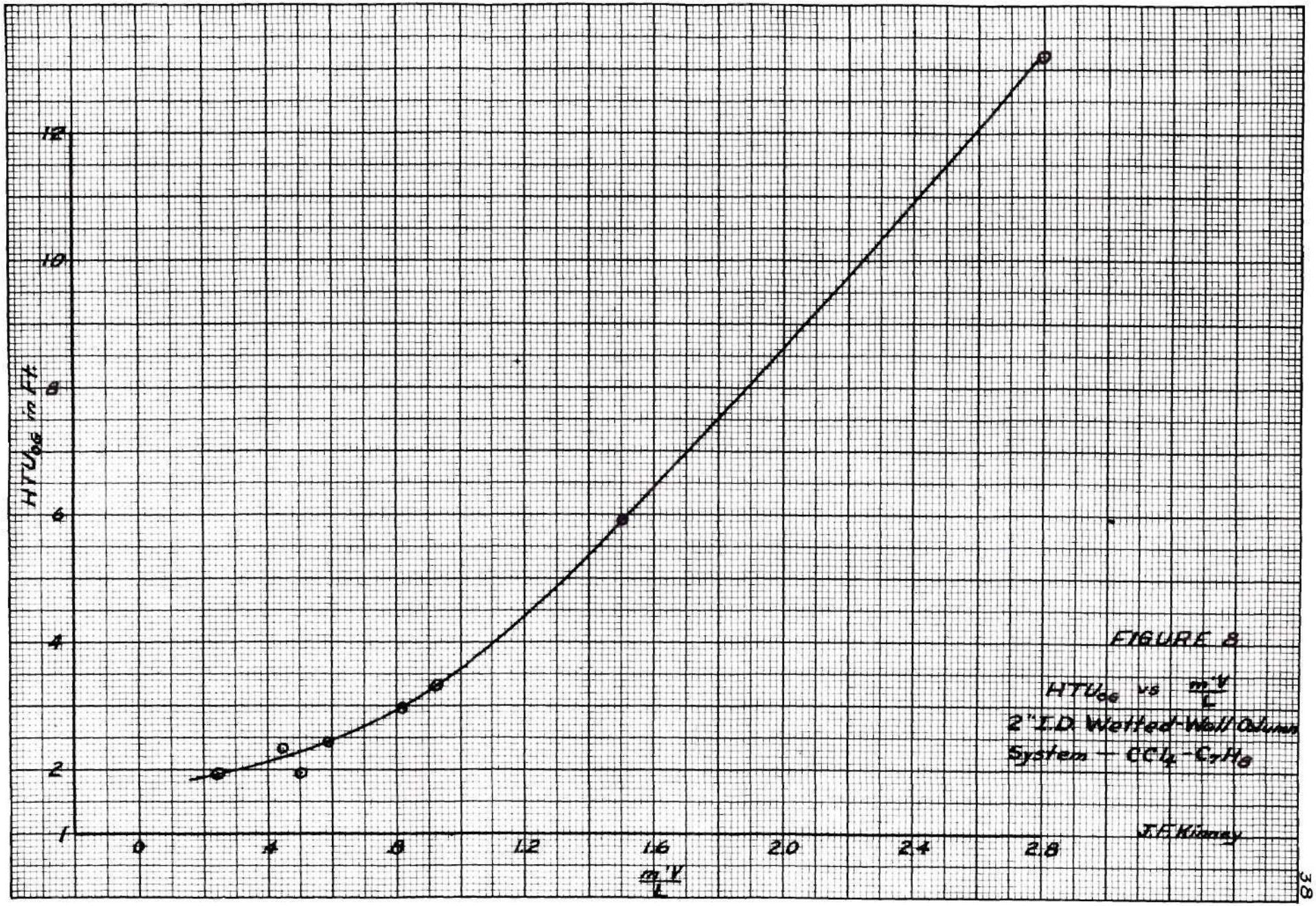
SUMMARY OF DATA

Run No.	28	29	31	32
V/L	0.469	0.521	1.0	1.0
N_{OG}	2.30	1.86	1.51	1.35
N_{OL}	1.01	0.901	1.09	1.13
HTU_{OG}(ft)	1.96	2.42	2.97	3.34
HTU_{OL}(ft)	4.44	4.99	4.14	3.98
m_{av}	0.781	0.764	0.602	0.642
m'	1.068	1.13	.822	.92
m"	.766	.797	.581	0.63
$\frac{m_{av}V}{L}$	0.367	0.398	0.602	0.642
$\frac{m'V}{L}$	.501	.589	.822	.92
$\frac{m_{av}V(HTU_{OL})}{L}$	1.62	1.84	2.49	2.55
HTU_{OG}/HTU_{OL}	.432	.485	.719	.841
$\frac{m'V}{L}HTU_{OL}$	2.22	2.94	3.40	3.66

TABLE I (CONT'D)

SUMMARY OF DATA

Run No.	33	36	37	38
V/L	0.545	0.331	1.596	2.97
N_{OG}	1.945	2.32	0.76	0.341
N_{OL}	0.882	0.599	0.939	0.755
HTU_{OG}(ft)	2.31	1.94	5.92	13.2
HTU_{OL}(ft)	5.11	7.38	4.79	5.96
m_{av}	0.604	0.616	0.664	0.612
m'	.82	.74	.943	.937
m"	.569	.581	.664	.681
$\frac{m_{av} V}{L}$	0.329	0.205	1.06	1.82
$\frac{m'V}{L}$	.446	.2445	1.505	2.785
$\frac{m_{av}V(HTU_{OL})}{L}$	1.68	1.51	5.08	10.85
HTU_{OG}/HTU_{OL}	.4525	.2585	1.235	2.22
$\frac{m'V}{L}HTU_{OL}$	2.285	1.81	7.21	16.6


HTU$_{OG}$ in Ft.
12
10
8
6
4
2
1
0
4
8
12
16
20
24
28
$\frac{m'V}{L}$
FIGURE 8
HTU$_{OG}$ vs $\frac{m'V}{L}$
2" I.D Wetted-Wall Column
System — CCl_4 - C_7H_8
J.F. Kinney

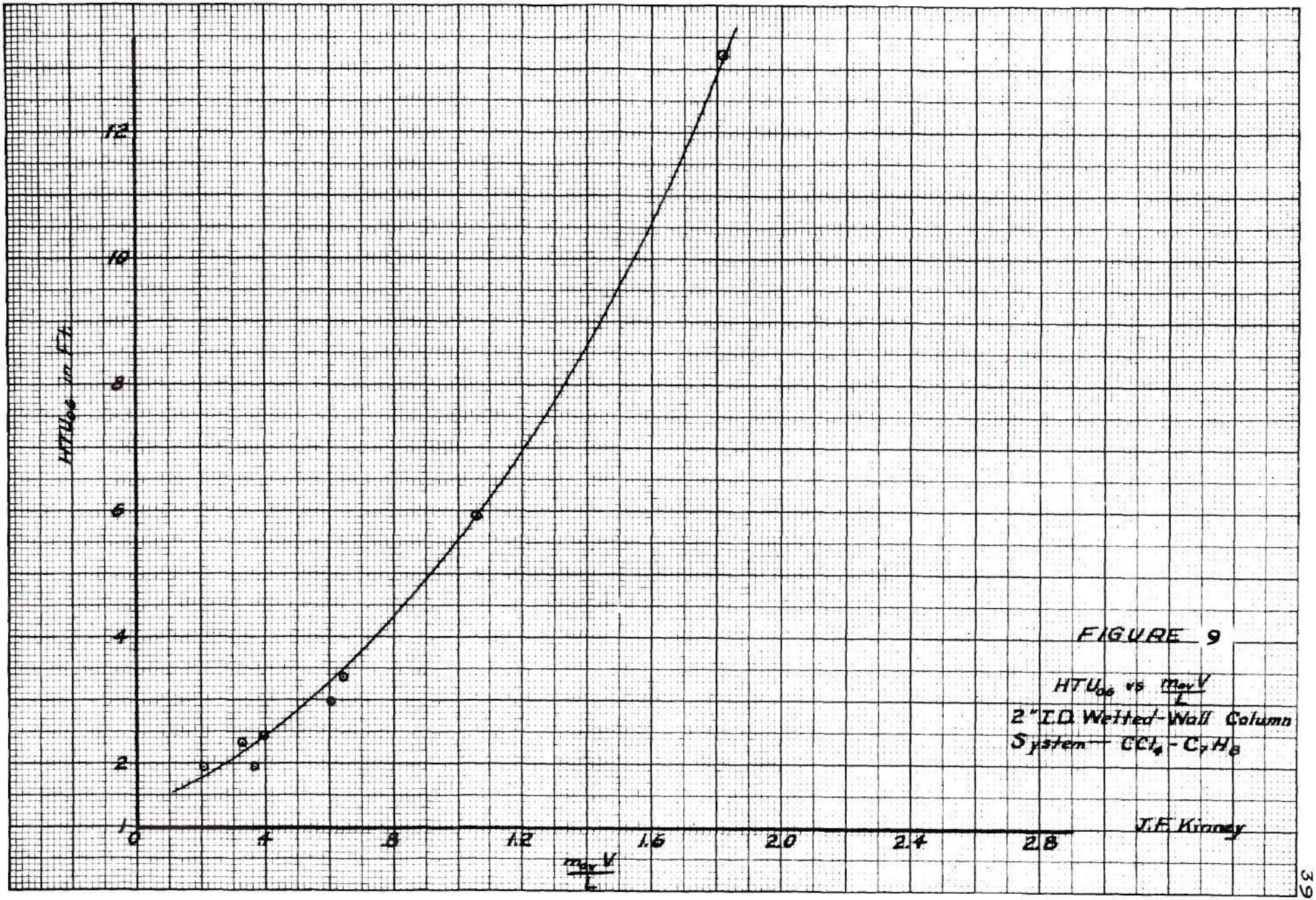


FIGURE 9

HTU_{OG} vs $\frac{m_{av}V}{L}$

2" I.D. Wetted-Wall Column

System — CCl_4 - C_7H_8

J.F. Kinney

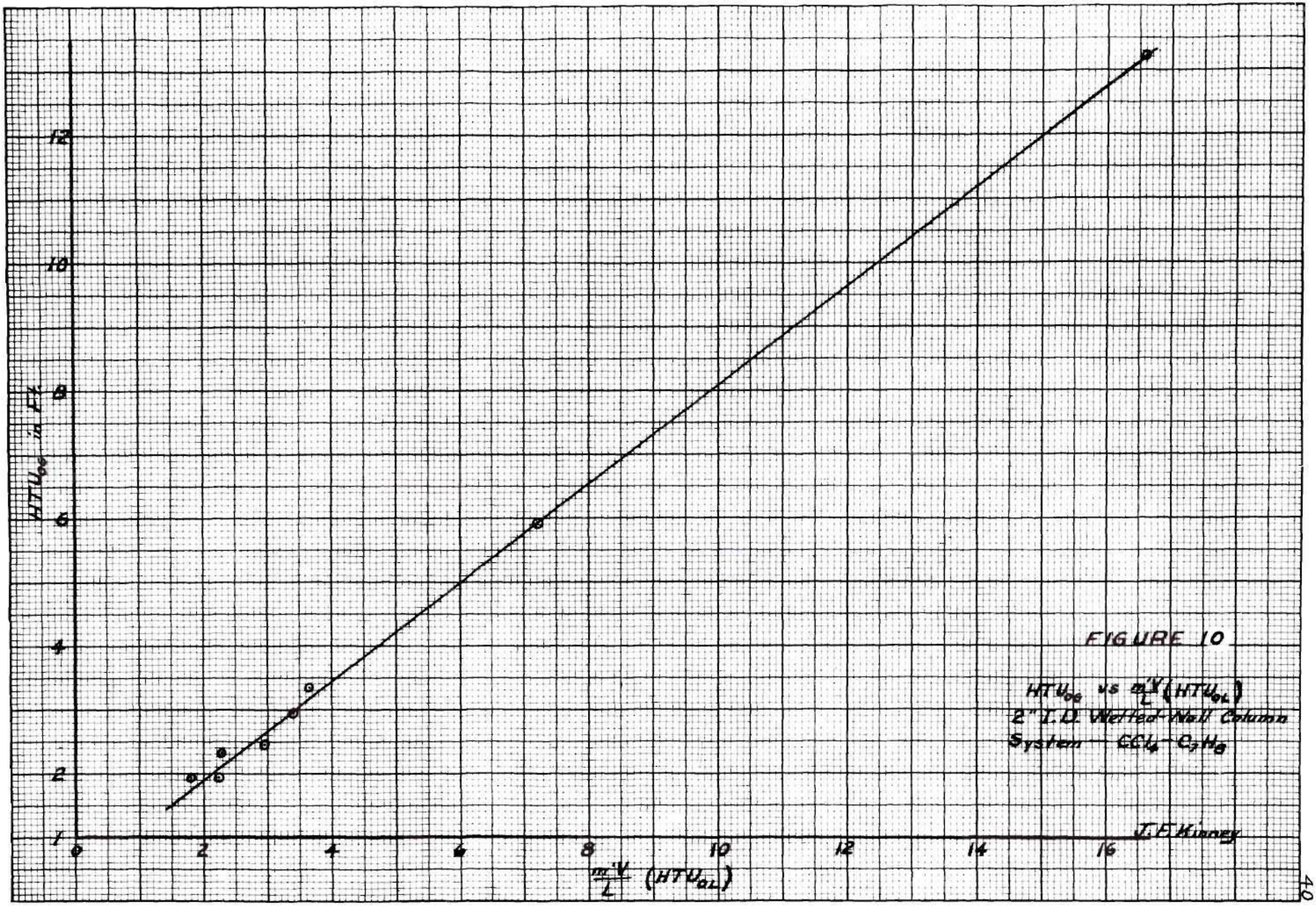


FIGURE 10

HTU_{OG} vs $\frac{m'V}{L}(HTU_{OL})$

2" I.D. Wetted-Wall Column

System — CCl_4 - C_3H_8

J. E. Kinney

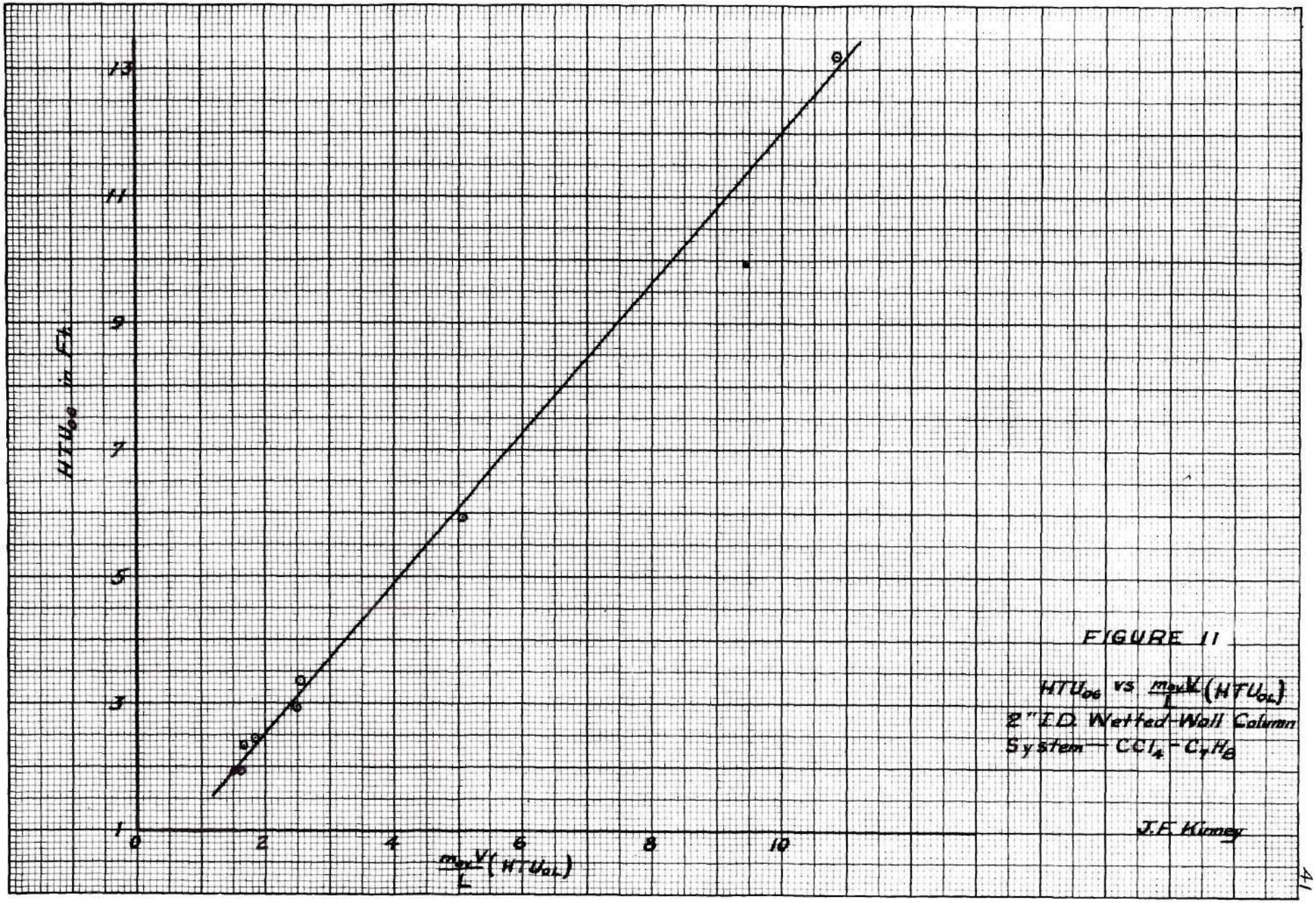


FIGURE 11

HTU_{OG} vs $\frac{m_{ex}V}{L}(HTU_{OL})$

2" I.D. Wetted-Wall Column

System — CCl_4 - C_7H_8

J.F. Kinney

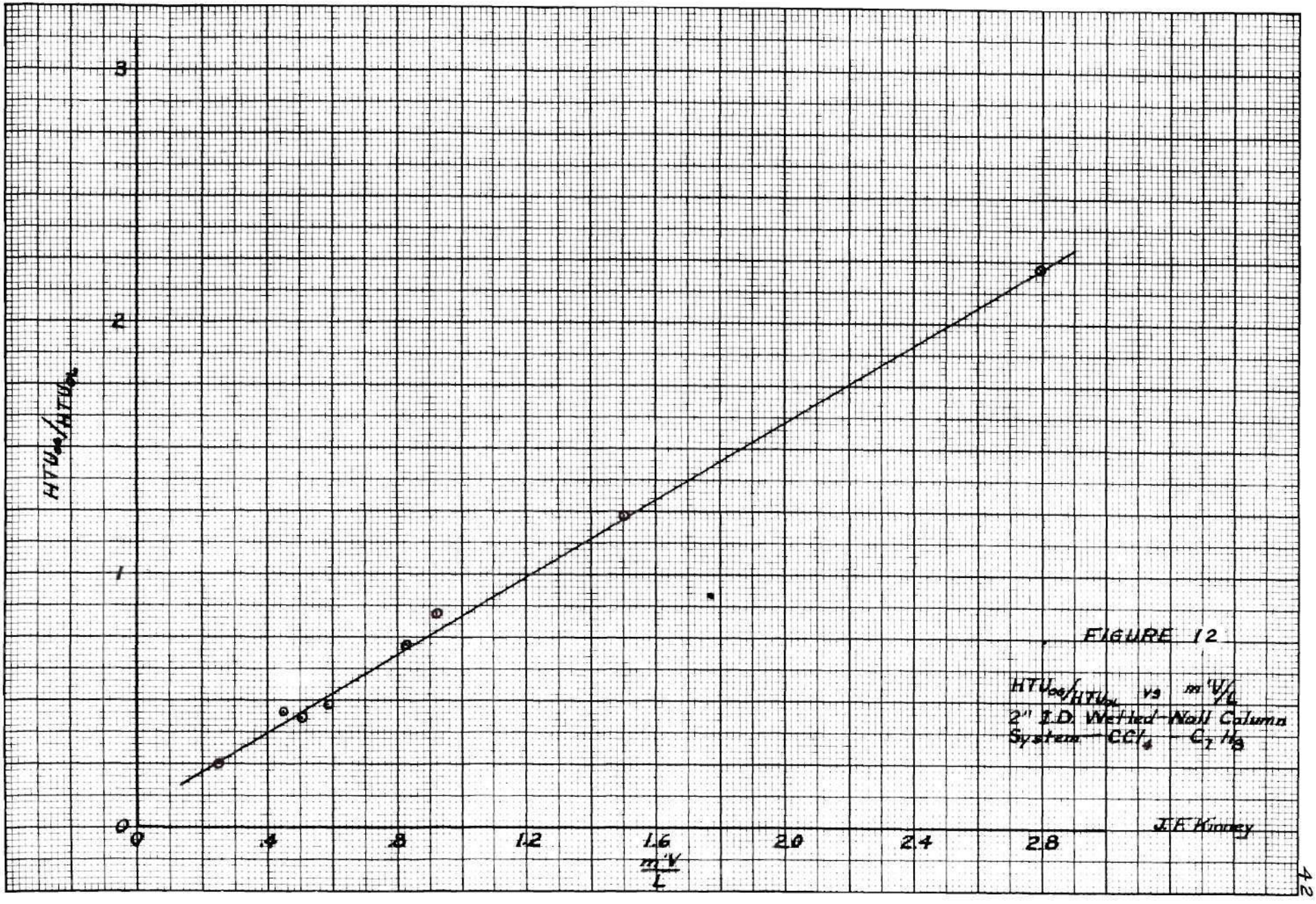


FIGURE 12

HTU_{oe}/HTU_{ox} vs $m'V/L$
2" I.D. Wetted-Wall Column
System — CCl_4 — C_7H_8

J.F. Kinney

DISCUSSION OF RESULTS

The primary object of this project was the construction of an equilibrium still, incorporating a wetted-wall column in which the reflux ratio V/L could be varied from zero to high values. Preliminary data were to be obtained to indicate that the equipment would be of use in further studies and evaluations of the transfer unit.

It should be stated here that the vapor velocities used were definitely in the transition region between streamline and turbulent flow. Consequently, the equations of Chilton and Colburn, and Johnstone and Pigford, would not apply. The equation of Gilliland and Sherwood is also inapplicable because of the flow rates involved.

It will be noticed that HTU_{OG} is not linear with $m_{av}V/L$ or $m'V/L$. This is believed to be due to the inconstancy of HTU_L.

The evaluation of m suggested by Furnas and Taylor, m_{av}, is compared with Lewis's m'. It is believed that m' smoothes out the data to a greater extent, that is, the scattering of points is somewhat less when using m' than when using m_{av}.

This point is brought out to a greater extent when the data of Deed, Schutz, and Drew (5), is plotted using m' as compared to the plot using m_{av}. The scattering of the points is decreased considerably. The HTU_{OG} versus $m'V/L$ plot reveals a smaller amount of curvature. This is noticeable with both sets of data but the data of Deed, Schutz, and Drew, brings out this straightening more noticeably.

The values of HTU_{OG} obtained by Deed, Schutz, and Drew, are somewhat lower than those obtained here. However, this is reasonable since they used a packed column where the interfacial area is considerably greater.

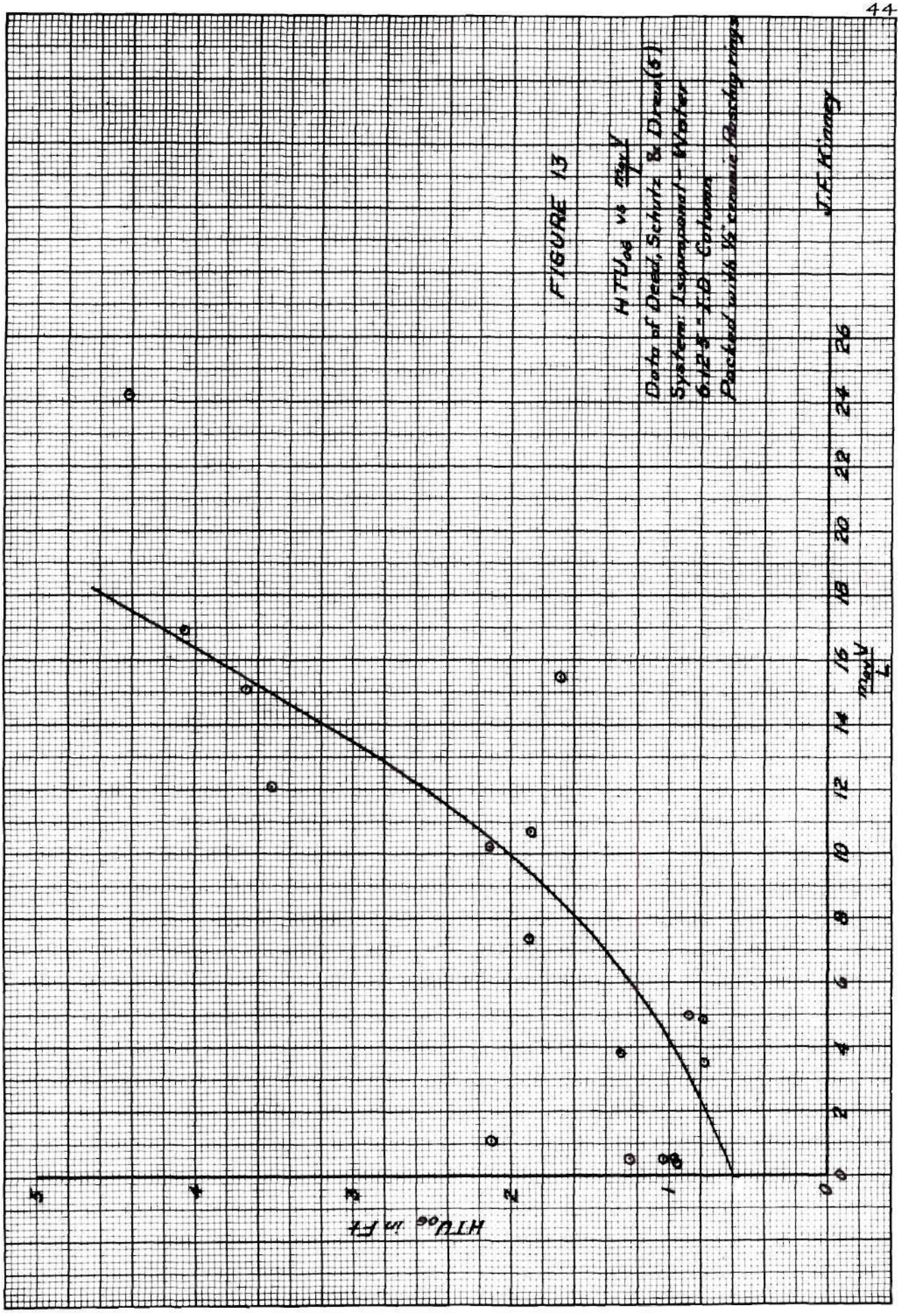


FIGURE 13
$H_{TU_{OG}}$ vs $\frac{1}{A_{meas}}$
Data of Deed, Schutz & Drexel(6)
System: Isopropanol - Water
6"ø - 5ft. Column
Packed with ½" ceramic Raschig rings
$\frac{1}{A_{meas}}$
$H_{TU_{OG}}$ in Ft
0
2
4
6
8
10
12
14
16
18
20
22
24
26
1
2
3
4
5

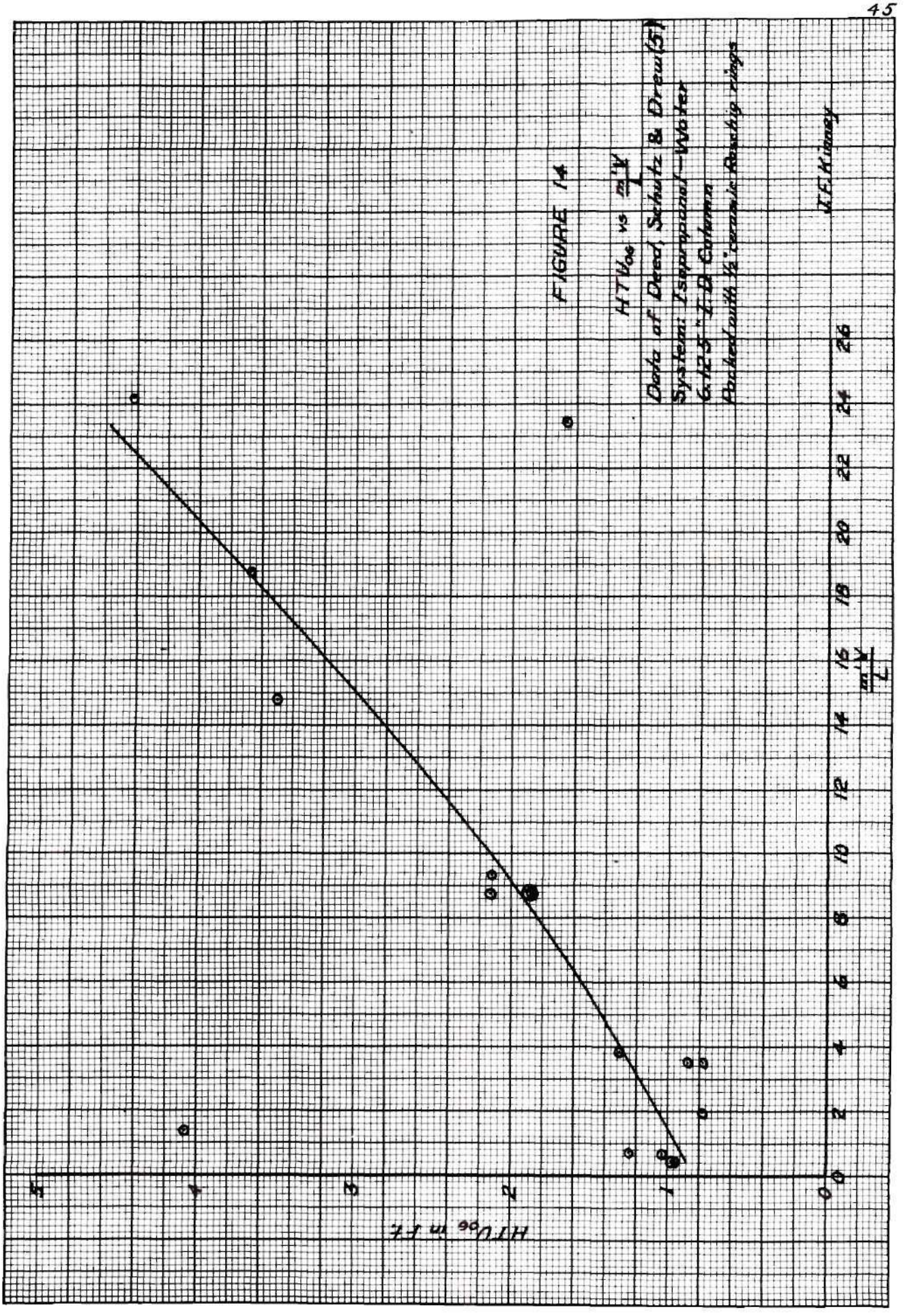


FIGURE 14
HTU$_{OE}$ vs $\frac{m}{L}$
Data of Deed, Schultz & Drew (5)
System: Isopropanol—Water
6.125" I.D. Column
Packed with ½" Ceramic Raschig rings
H.T.U. in ft
$\frac{m}{L}$
HTU$_{OE}$ in ft
0
2
4
6
8
10
12
14
16
18
20
22
24
26

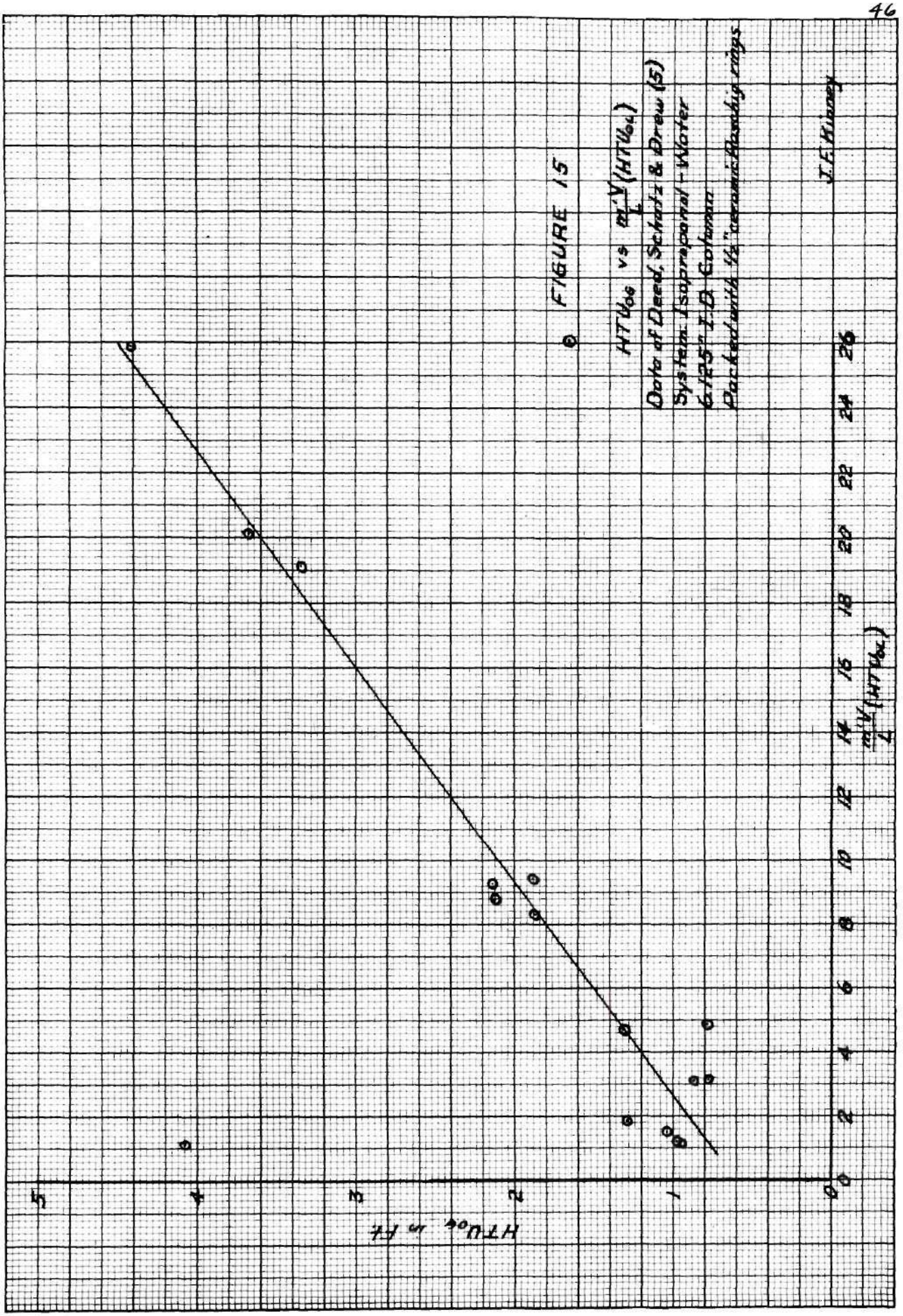

HTU$_{OL}$ in Ft
0
1
2
3
4
5
$\frac{m}{L}V_t$ (HTU$_{OL}$)
0
2
4
6
8
10
12
14
16
18
20
22
24
26
HTU$_{OL}$ vs $\frac{m}{L}V_t$ (HTU$_{OL}$)
Data of Deed, Schultz & Drew (5)
System: Isopropanol - Water
6"x25" I.D. Column
Packed with ½" ceramic Raschig rings
J.F. Kinney

FIGURE 15

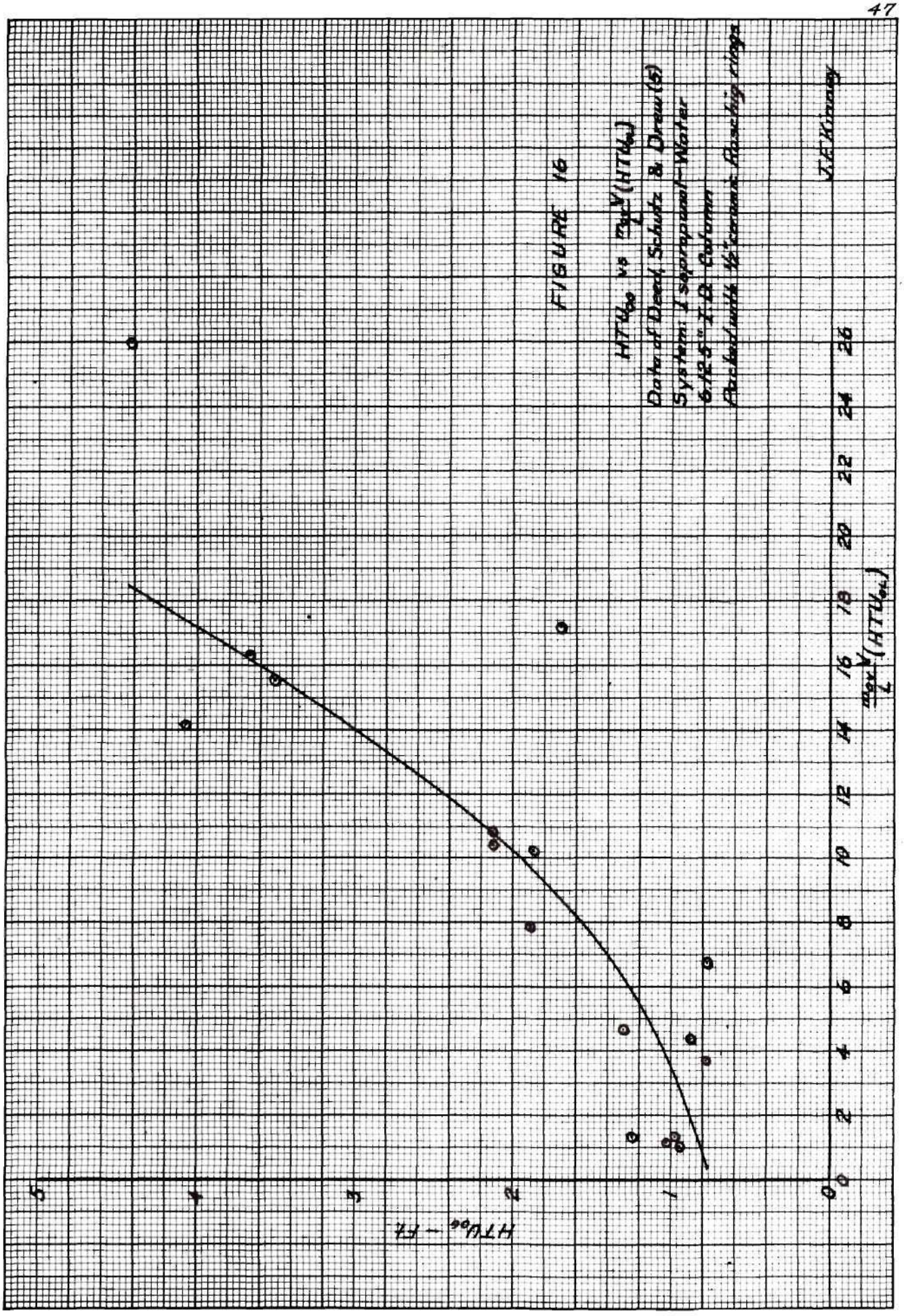


FIGURE 16
HTU_{oo} vs $\frac{1}{m_{av}}V(HTU_{oo})$
Data of Deed, Schutz & Drew(6)
System: Isopropanol-Water
6.125" I.D. Column
Packed with ½" ceramic Raschig rings
T.F. Hixson
$HTU_{oo} - H$
$\frac{1}{m_{av}}V(HTU_{oo})$

TABLE II

SUMMARY OF DATA OF DEED, SCHUTZ, AND DREW

Run No.	35	37	38	39
V/L	1	1	1	1.209
HTU_{OG}(ft)	0.955	0.787	0.784	4.07
HTU_{OL}(ft)	2.54	1.376	1.62	0.837
m'	0.451	3.54	1.95	11.3
m''	.363	.2385	.207	0.346
m_{av}	0.40	4.92	3.52	14.0
$\frac{m'V}{L}$	.451	3.54	1.95	1.368
$m_{av}\ V/L$	.40	4.92	3.52	16.95
$\frac{m'V(HTU_{OL})}{L}$	1.145	4.87	3.16	1.145
$\frac{m_{av}V\ H_{OL}}{L}$	1.016	6.78	3.70	14.18

TABLE II (CONT'D)

SUMMARY OF DATA OF DEED, SCHUTZ, AND DREW

Run No.	41	43	45	46
V/L	1.13	1.112	1.418	1.62
HTU_{OG}(ft)	1.875	2.125	1.035	0.871
HTU_{OL}(ft)	0.954	0.939	2.145	0.879
m'	7.75	8.425	0.501	2.19
m"	0.3585	0.371	0.332	0.271
m_{av}	9.50	9.96	0.38	3.09
$\frac{m'V}{L}$	8.75	9.36	.71	3.55
m_{av} V/L	10.73	1.108	0.538	5.0
$\frac{m'V(HTU_{OL})}{L}$	8.34	8.78	1.523	3.12
$\frac{m_{av}V}{L}$ H_{OL}	10.25	10.4	1.157	4.39

TABLE II (CONT'D)

SUMMARY OF DATA OF DEED, SCHUTZ, AND DREW

Run No.	47	49	53	56
V/L	1.862	2.04	1.462	1.329
HTU_{OG}(ft)	1.888	3.67	1.246	2.13
HTU_{OL}(ft)	1.069	1.073	2.56	1.059
m'	4.73	9.21	0.504	6.64
m"	0.275	0.343	0.317	0.32
m_{av}	3.95	7.44	0.36	7.71
$\frac{m'V}{L}$	8.8	18.78	.737	8.75
m_{av} V/L	7.35	15.17	.527	10.23
$\frac{m'V}{L}(HTU_{OL})$	9.42	20.15	1.887	9.27
$\frac{m_{av}V}{L} H_{OL}$	7.85	16.3	1.345	10.85

TABLE II (CONT'D)

SUMMARY OF DATA OF DEED, SCHUTZ, AND DREW

Run No	61	62	68	71	72
V/L	2.92	3.02	1.70	2.325	2.14
HTU_{OG}(ft)	1.31	3.515	0.974	1.69	4.42
HTU_{OL}(ft)	1.237	1.29	2.535	1.11	1.074
m'	1.765	4.91	0.288	10.09	11.3
m"	.209	0.27	0.3625	0.322	.421
m_{av}	1.32	4.00	0.34	6.67	11.3
$\frac{m'V}{L}$	3.82	14.82	0.49	23.45	24.2
m_{av} V/L	3.86	12.08	0.588	15.5	24.2
$\frac{m'V}{L}(HTU_{OL})$	4.73	19.1	1.242	26.05	25.9
$\frac{m_{av}V\ H_{OL}}{L}$	4.67	15.58	1.36	17.2	26.0

RECOMMENDATIONS

Experimentally, the operation of the still is quite satisfactory. The time requirement for the approach to equilibrium is approximately four hours. However, this could be decreased to some extent with properly designed changes. The boiler could be increased in size, which would decrease the concentration change due to hold-up. Also the increased size would enable the use of a larger size (and hence an increased wattage) GlasCol heating mantle. The increased heat-flux available with the larger mantle would provide for the use of higher rates of flow within the column. (The mass rate of flow in this column is strictly limited by the size of the base heater. The flow rates within the column are definitely in the transition region between streamline and turbulent flow.)

The flowmeters, while providing the least change of flowmeter calibration with changes in concentration, have disadvantages. The small size of the orifice serves as a trap for foreign matter, some of which is unavoidable within the system. Any foreign matter in the orifice is detectable by a change in the flow pattern from the orifice. However, the lodging of any matter in the orifice after several hours of operation is particularly objectionable since it greatly delays the approach to equilibrium. In order to prevent a shutdown at such a time, a soft copper wire was designed to dislodge the obstruent material. However, it was learned that one of the flowmeter calibrations changed (increased) after many applications of the soft copper wire instrument, that is, the orifice diameter was increased slightly. This disadvantage, in all probability, will not be so noticeable at larger rates of flow where the orifice diameter will be increased. However, this flowmeter is practically independent, volumetrically, of the density and

of the viscosity of the flowing liquid. This type of meter also affords complete visibility during operation.

The thermocouples were kept small and used no protective shield in order to keep the time lag short. A snugly-fitting glass shield would probably not increase this time lag appreciably and would incorporate into the thermocouples increased life and probably increased accuracy.

Should it be desired to operate the column as a packed tower, the installation of packing in the column could be accomplished relatively easily with the exception of providing a liquid distributor at the top. Prehaps a perforated plate with a small head of liquid above the plate would accomplish equal liquid distribution. However, the installation of the plate requires the installation of a large spherical joint in the column wall thus enabling the top of the column to be removed at will and providing an opening the size of the inside diameter of the column for insertion of the plate.

TABLE OF NOMENCLATURE

A	Interface area, sq. ft.
B_L	$\left(\frac{3\mu_L L}{\rho_L^2 g}\right)^{1/3}$ Nusselt expression for laminar film thickness, ft.
D	diffusivity, sq. ft./hr.
E	percentage error in heat balance
G	mass rate of vapor flow, lb./(hr.) (sq. ft.)
H_t	HTU height of a transfer unit, ft
K	overall gas film absorption coefficient, lb. mols./(atm.) (hr.) (sq. ft.)
L	molal rate of liquid flow, lb. mols./hr.
L'	L/Area = lb. mols./sq. ft. - hr.
$\overline{L}$	mass rate of liquid flow, lb./(hr.) (sq. ft.)
M	molecular weight of more volatile component
M_m	average molecular weight of vapor stream
M_L	average molecular weight of liquid stream
N_t	number of transfer units
O	- P molal rate of feed, lb. mols./hr
S	cross sectional area over which G is measured, sq. ft.
V	molal rate of gas flow, lb. mols./hr.
V'	V/Area= lb. mols./(hr.) (sq. ft.)
W	rate of diffusional mass transfer, lb. mols./hr.
Z	height of packed section, ft.
a	surface area of packing per unit of packed volume, sq. ft./cu. ft.
d	differential operator
d_t	diameter of column, ft.

g accelaration of gravity, ft./hr./hr.

k_G individual gas film mass transfer coefficient, lb. mols./(atm.) (hr.) (cu. ft. of packed section.)

k_L individual liquid film mass transfer coefficient, lb. mols./(atm.) (hr.) (cu. ft. of packed section)

m $\dfrac{y^* - y}{x - x^*}$

m_{av} $\dfrac{\left[\int_0^{y_2^*} m dy\right] - \left[\int_0^{y_1^*} m dy\right]}{y_2^* - y_1^*}$

$\overline{m}$ $\dfrac{HTU_{OG}}{HTU_{OL}} \cdot \dfrac{L}{V}$

m_L $\dfrac{y^* - y_i}{x - x_i}$

m_G $\dfrac{y_i - y}{x_i - x^*}$

p partial pressure of diffusing component, atm.

p^* equilibrium partial pressure of diffusing component out of liquid, atm.

Δp $p - p^*$

u average stream velocity, ft./sec.

x mole fraction more volatile component in liquid

x^* equilibrium mole fraction more volatile component in liquid

x_o mole fraction more volatile component in liquid feed

x_p mole fraction more volatile component in overhead liquid product

x_w mole fraction more volatile component in liquid in boiler

y mole fraction more volatile component in vapor

y^* equilibrium mole fraction more volatile component in vapor

Δy $y^* - y$

α	a constant		
β	a constant		
Γ	peripheral rate of liquid flow, lb./(hr.) (ft. of wetted perimeter)		
μ	viscosity of stream, lb./(ft.) (hr.)		
ρ	density of stream, lb./cu. ft.		
Re_G	$\frac{d_t u_G \rho_G}{\mu_G}$	Reynolds number	any consistent units
Sc_G	$\frac{\mu_G}{\rho_G D_G}$	Schmidt number	any consistent units

Subscripts

G	refers to the gas stream
m	mean value over the column height
L	refers to the liquid stream
OG	refers to overall condition based on the gas stream
OL	refers to overall conditions based on the liquid stream
1	refers to conditions at the bottom of the column
2	refers to conditions at the top of the column
i	refers to conditions at the vapor-liquid interface

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APPENDIX I

TABLE III

DATA

Run No.	28	29	31	32
V	.0221	.0237	.03	.046
V'	1.013	1.087	1.375	2.11
G	139.4	147.8	196	294.5
L	.0471	.0455	.03	.046
Γ	11.85	11.48	8.17	12.29
y_1	0.3525	0.343	0.612	0.5675
Temp of y_1	198 F	205 F	204 F	207 F
y_2	0.742	0.7125	0.8225	0.7775
Temp of y_2	183 F	189 F	177 F	181 F
x_2	0.643	0.648	0.8225	0.7775
Temp of x_2	175 F	174 F	75 F	158 F
x_1	0.446	0.4575	0.6125	0.540
Temp of x_1	191 F	196 F	185 F	191 F
E	5.91	1.23	15.8	0.55
V/L	0.469	0.521	1.00	1.00
N_{OL}	1.013	0.901	1.087	1.13
N_{OG}	2.295	1.86	1.513	1.348
Z	4.5	4.5	4.5	4.5

TABLE III (CONT'D)

DATA

Run No.	33	36	37	38
V	0.0259	.0243	.0414	
V'	1.188	1.113	1.9	
G	170.8	112.0	162.3	
L	.0476	.0514	.0259	
Γ	12.83	13.39	2.73	
y_1	0.548	0.6465	0.616	0.6475
Temp of y_1	195 F	194 F	201 F	199 F
y_2	0.8375	0.838	0.725	0.6925
Temp of y_2	174 F	190 F	196 F	193 F
x_2	0.800	0.728	0.725	0.6925
Temp of x_2	154 F	189 F	175 F	171 F
x_1	0.594	0.6425	0.556	0.564
Temp of x_1	186 F	172 F	198 F	195 F
E	6.87	9.92	0.97	
V/L	0.545	0.331	1.596	2.97
N_{OL}	0.882	0.599	0.939	0.755
N_{OG}	1.945	2.32	0.76	0.34
Z	4.5	4.5	4.5	4.5

TABLE III (CONT'D)

DATA

Run No.	28	29	31	32
HTU_{OG}(ft)	1.962	2.42	2.97	3.34
HTU_{OL}(ft)	4.44	4.99	4.14	3.98
m_{av}	0.781	0.764	0.602	0.642
$\frac{m_{av}V}{L}$	0.367	0.398	0.602	0.642
$\frac{mdy}{y}$	2.45	2.1	1.245	1.239
m^1	1.068	1.13	.822	.92
$\frac{dx}{x}$	1.324	1.13	1.872	1.795
m"	0.766	0.797	.581	0.63
$\frac{m'V}{L}$	.501	.589	.822	.92
$\frac{m'V}{L}(HTU_{OL})$	2.22	2.94	3.4	3.66
$\frac{m_{av}V}{L}(HTU_{OL})$	1.62	1.84	2.49	2.55
$\frac{HTU_{OG}}{HTU_{OL}}$	.432	.485	.719	.841

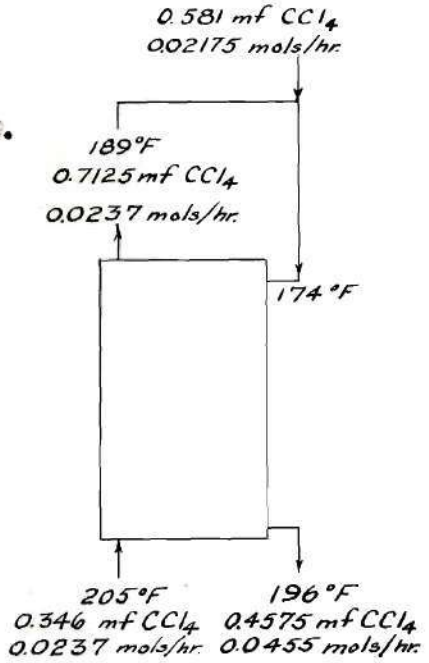
TABLE III (CONT'D)

DATA

Run No.	33	36	37	38
HTU_{OG}(ft)	2.31	1.94	5.92	13.22
HTU_{OL}(ft)	5.11	7.51	4.79	5.96
m_{av}	0.604	0.616	0.664	0.612
$\frac{m_{av}V}{L}$	0.329	0.205	1.058	1.82
$\frac{mdy}{y}$	1.58	1.716	.717	.319
m^1	.819	.74	.943	.937
$\frac{dx}{x}$	1.55	1.032	1.415	1.108
m"	.569	.581	.664	.681
$\frac{m'V}{L}$	.446	.2445	1.505	2.795
$\frac{m'V}{L}(HTU_{OL})$	2.285	1.81	7.21	16.6
$\frac{m_{av}V}{L}(HTU_{OL})$	1.68	1.51	5.08	1.085
$\frac{HTU_{OG}}{HTU_{OL}}$	.4525	.2585	1.235	2.22

APPENDIX II

SAMPLE CALCULATIONS



The calculations are performed for Run No. 29.

1. The temperature, concentration, and flow rate data may be shown diagramatically, as at the right:

2. The concentration of the reflux fed to the column is:

$$\frac{.02175(.581) + .0237(.7125)}{.0455}$$

$$= 0.648 \text{ mf } CCl_4.$$

3. The heat balances were calculated from total heat content charts (Figures 17, and 18) constructed from the data of the following:

Specific heats of liquids (14, 24)

Latent heats of vaporization (24)

The enthalpy of the saturated vapor is substantially linear with molar concentration and was so assumed.

$$.0237\ \left[.346(17{,}950) + .654(21{,}080)\right] + \left[.648(4{,}580) + .352(5{,}400)\right] .0455$$

$$= .0237\ \left[.7125(17650) + .2875(20{,}650)\right] + .0455\ \left[.4575(5350) + .5425(6{,}300)\right].$$

$$694 \doteq 703$$

$$\% \text{ error} = \frac{100(9)}{694} = 1.23.$$

4. A CCl_4 balance over the top portion of the column yields

$$Lx = Vy + Ox_o$$

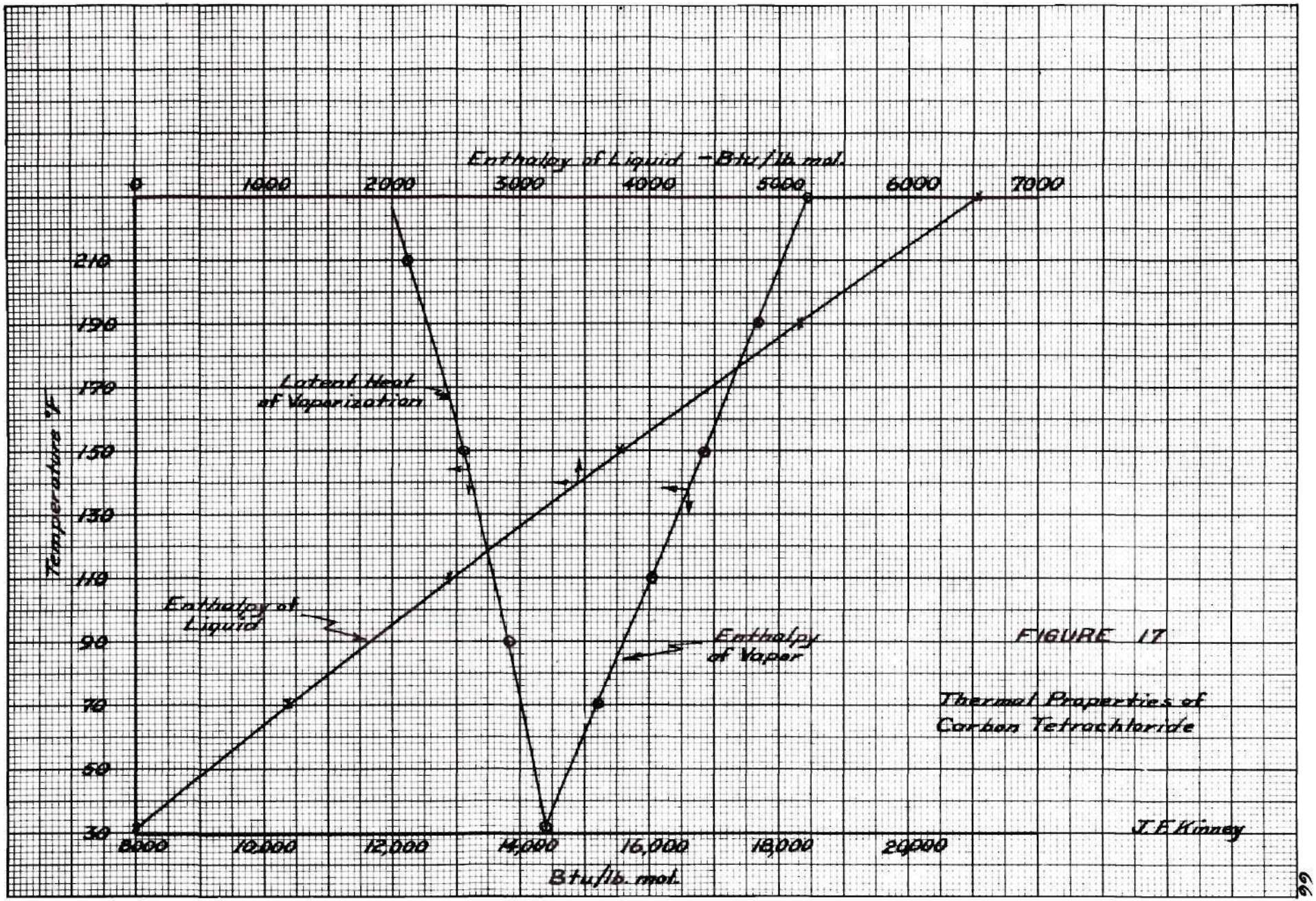

Enthalpy of Liquid → Btu/lb. mol.
0
1000
2000
3000
4000
5000
6000
7000
210
190
170
150
130
110
90
70
50
30
Temperature °F
Latent Heat of Vaporization
Enthalpy of Liquid
Enthalpy of Vapor
8000
10,000
12,000
14,000
16,000
18,000
20,000
Btu/lb. mol.
FIGURE 17
Thermal Properties of Carbon Tetrachloride
J. E. Kinney

FIGURE 18

Thermal Properties of Toluene

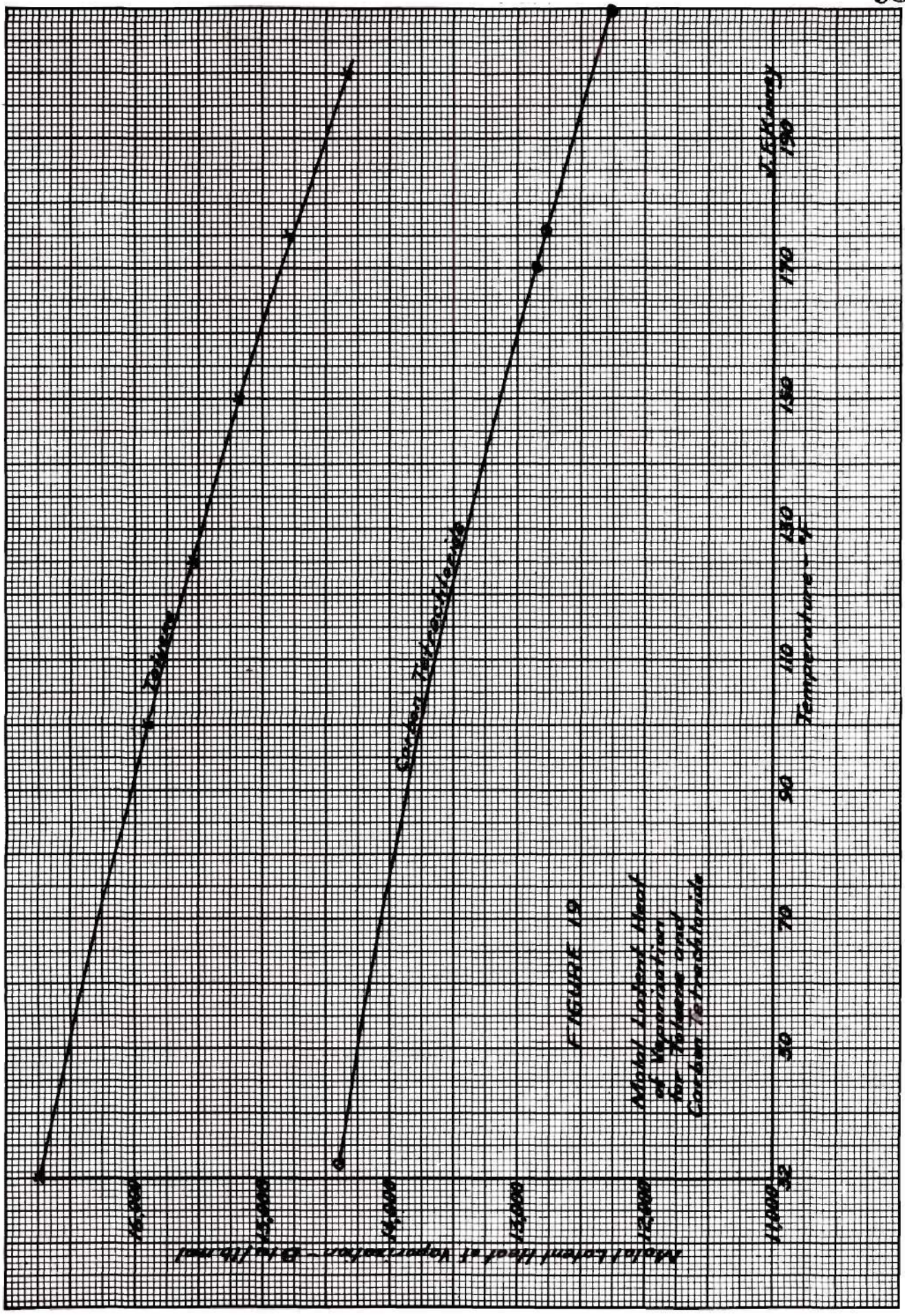

Molal Latent Heat at Temperature - B.t.u. per lb.mol
Temperature - °F
Efficiency
Sulfur Dioxide
Carbon Tetrachloride
Molal Latent Heat of Vaporization for Toluene and Carbon Tetrachloride

FIGURE 19

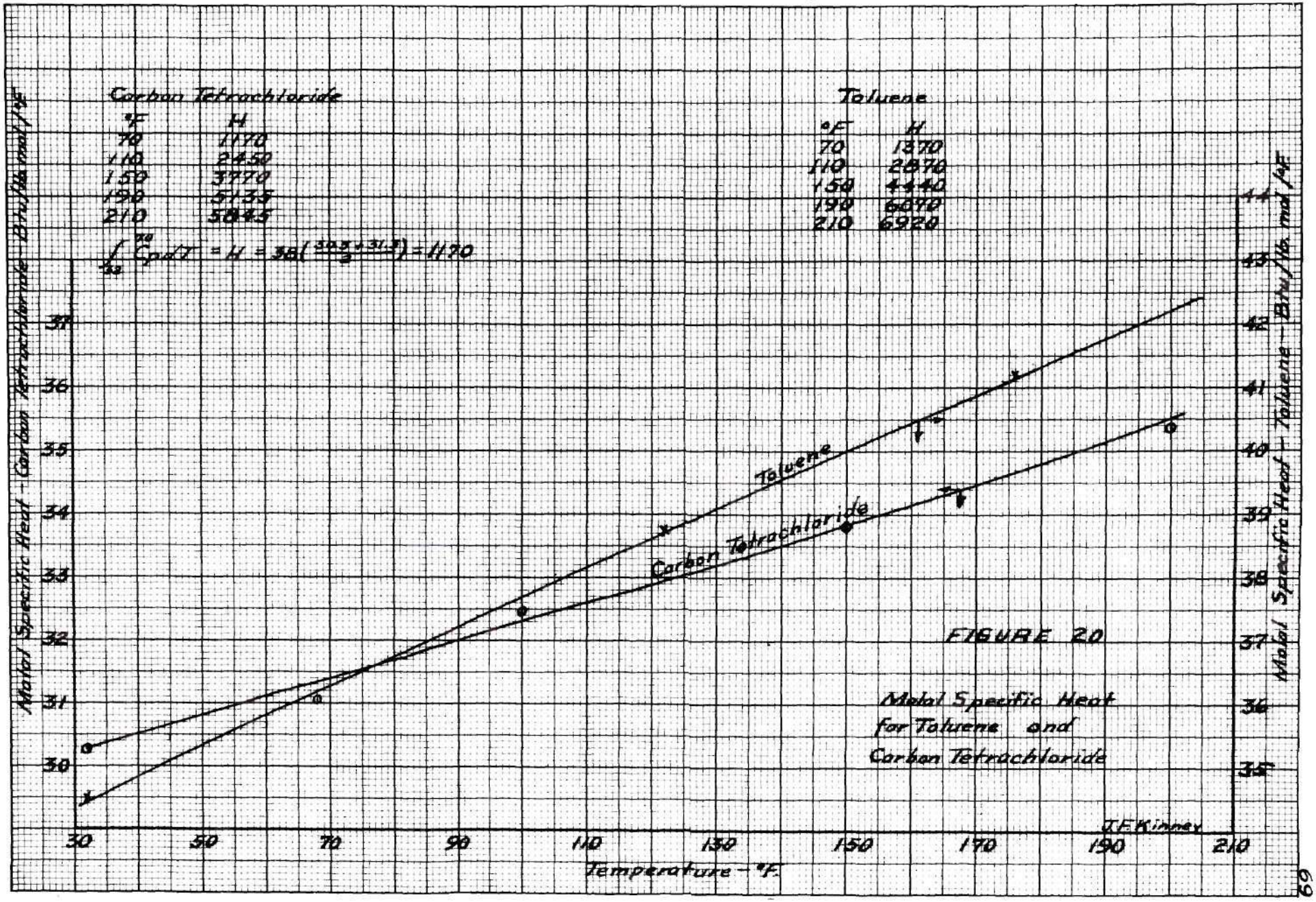


FIGURE 20

Molal Specific Heat
for Toluene and
Carbon Tetrachloride

or rearranging, the equation of the operating line is

$$y = \frac{L}{V} x - \frac{O}{V} x_o.$$

Substituting $L = .0455$ $V = .0237$

$O = .02175$ $x_o = .581$

$$y = \frac{.0455}{.0257} - \frac{.02175}{.0257}(.581)$$

$$= 1.92x - .534.$$

This operating line is drawn on the x vs y diagram (Figure 21).

5.- The graphical integration was carried out by the four-point Gauss method (11, 20).

$$N_{OG} = \int_a^b \frac{dy}{\Delta y} = (b-a)\left[.1739\left(\frac{1}{\Delta y_1} + \frac{1}{\Delta y_4}\right) + .3261\left(\frac{1}{\Delta y_2} + \frac{1}{\Delta y_3}\right)\right].$$

where $\Delta y_1 = y_1^* - y_1$

$\Delta y_2 = y_2^* - y_2$

and $y_1 = a + (b-a)\ (.0694)$

$y_2 = a + (b-a)\ (.33)$

$y_3 = a + (b-a)\ (.67)$

$y_4 = a + (b-a)\ (.931).$

These operations are:

$(b-a) = .7125 - .343 = .3695$

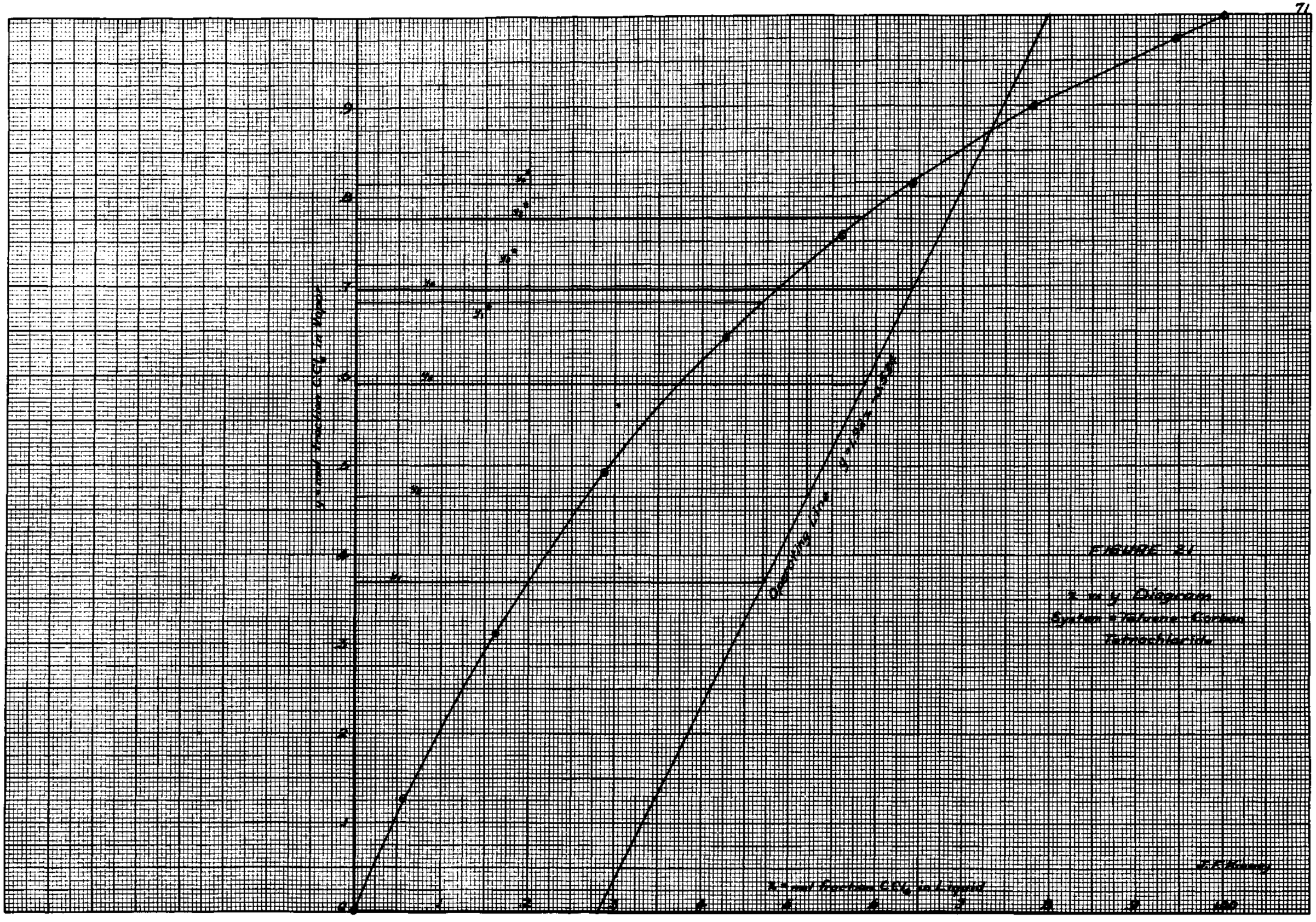


FIGURE 21

x vs y Diagram
System Toluene Carbon
Tetrachloride

E.F.Kenny

		y	y*	y* - y
y_1 =	.343	.3695(.0694) = .3687	.6825	.3139
y_2 =	.343	.3695(.33) = .465	.7245	.2595
y_3 =	.343	.3695(.67) = .5905	.7745	.184
y_4 =	.343	.3695(.931) = .697	.8075	.1205

$$N_{OG} = .3695\left[.1739\left(\frac{1}{.3139}+\frac{1}{.1205}\right)+.3261\left(\frac{1}{.2595}+\frac{1}{.184}\right)\right]$$

$$= 1.86$$

6. $$HTU_{OG} = \frac{Z}{N_{OG}} = \frac{4.5}{1.86} = 2.42 \text{ ft.}$$

7. The average slope of the equilibrium curve was read from the integrated curve (Figure 22) of the slope of the equilibrium curve (Figure 23), thus,

at $y = .7125$ $y^* = .816$ $\int_o^2 mdy = 1.195$

at $y = .343$ $y^* = \frac{.6715}{.1445}$ $\int_o^1 mdy = \frac{1.082}{0.113}$

$$m_{av} = \frac{\left[\int_0^2 mdy\right] - \left[\int_0^1 mdy\right]}{y_2^* - y_1^*} = \frac{.113}{.1445} = .782$$

8. HTU_{OL} was calculated in the following manner, using the Gauss method of graphical integration:

$$(b-a) = .648 - .4575 = .1905$$

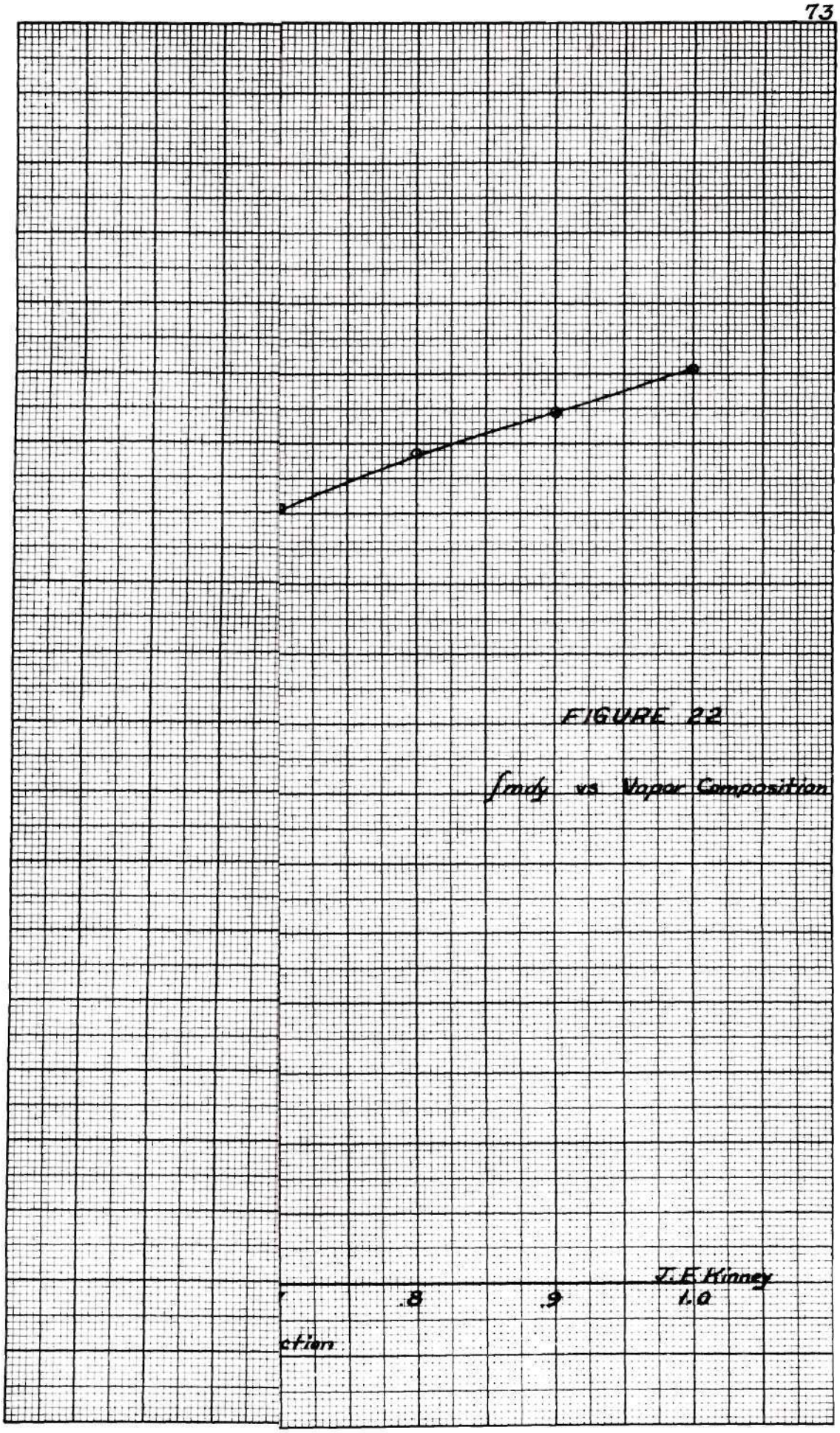


FIGURE 22

f_{mdy} vs Vapor Composition

y	Area	f_m / Σ
0		
.1	.244	0.2
.2	.195	0.4
.3	.171	0.6
.4	.153	0.7
.5	.133	0.8
.6	.115	1.01
.7	.0997	1.10
.8	.078	1.18
.9	.059	1.24
1.0	.063	1.30

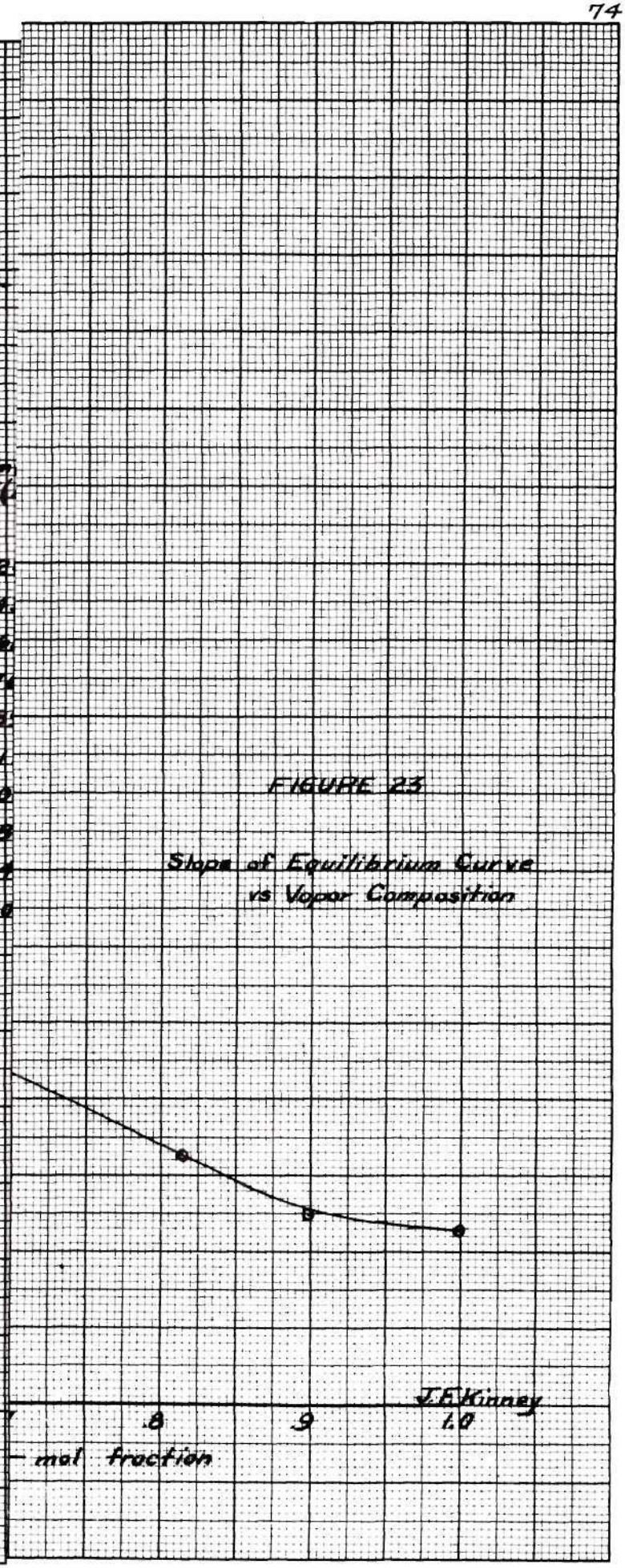


FIGURE 25

Slope of Equilibrium Curve vs Vapor Composition

T.E. Kinney

		x	x*	Δx	$1/\Delta x$
x_1 = .4575	.1905(.0694)	= .4707	.199	.2717	4.685
x_2 = .4575	.1905(.33)	= .5203	.2675	.2528	3.955
x_3 = .4575	.1905(.67)	= .5852	.3725	.2127	4.70
x_4 = .4575	.1905(.931)	= .6348	.475	.1598	6.26

$$N_{OL} = .1905 \left[.1739(10.95) + .3261(8.66)\right] = .901$$

$$HTU_{OL} = \frac{H}{N} \frac{4.5}{.901} = 4.99 \text{ ft.}$$

9. m' was calculated in the following manner, using the Gauss method of graphical integration:

From calculation No. 5, and the graph of m vs y (Figure 23),

	y	Δy	m	$m/\Delta y$
y_1 =	.3687	.3139	1.49	4.75
y_2 =	.465	.2595	1.305	5.04
y_3 =	.5905	.184	1.08	5.87
y_4 =	.687	.1205	.900	7.47

$$\int \frac{mdy}{\Delta y} = 0.3695 \left[.1739(12.22) + .3261\ (10.91)\right]$$

$$= .3695\ (5.68) = 2.1$$

$$\int \frac{dy}{\Delta y} = N_{OG} = 1.86$$

$$m' = \frac{\int \frac{mdy}{\Delta y}}{\int \frac{dy}{\Delta y}} = \frac{2.1}{1.86} = 1.13$$

10. m" was calculated in the following manner, using the Gauss method

of graphical integration:

From calculation No. 8, - and graph of m vs x (Figure 24)

	x	Δx	m	$1/m\Delta x$
x_1 =	.4707	.2717	.915	4.025
x_2 =	.5203	.2528	.84	4.71
x_3 =	.5852	.2127	.74	6.35
x_4 =	.6348	.1598	.665	9.41

$$\int \frac{dx}{m\Delta x} = 0.1905 \left[.1739(13.44) + .3261(11.06)\right]$$

$$= .1905(5.945) = 1.13$$

$$\int \frac{dx}{\Delta x} = N_{OL} = 0.901$$

$$m'' = \frac{\int \frac{dx}{\Delta x}}{\int \frac{dx}{m\Delta x}} = \frac{0.901}{1.13} = 0.797$$

11. $\frac{m'V}{L} = 1.13 \times 0.521 = 0.589$

12. $\frac{m_{av}V}{L} = .782 \times .521 = 0.398$

13. $\frac{m'V}{L}(HTU_{OL}) = 0.589 \times 4.99 = 2.22$

14. $m_{av}V(HTU_{OL}) = .398 \times 4.99 = 1.84$

15. $\frac{HTU_{OG}}{HTU_{OL}} = \frac{2.42}{4.99} = 0.485$

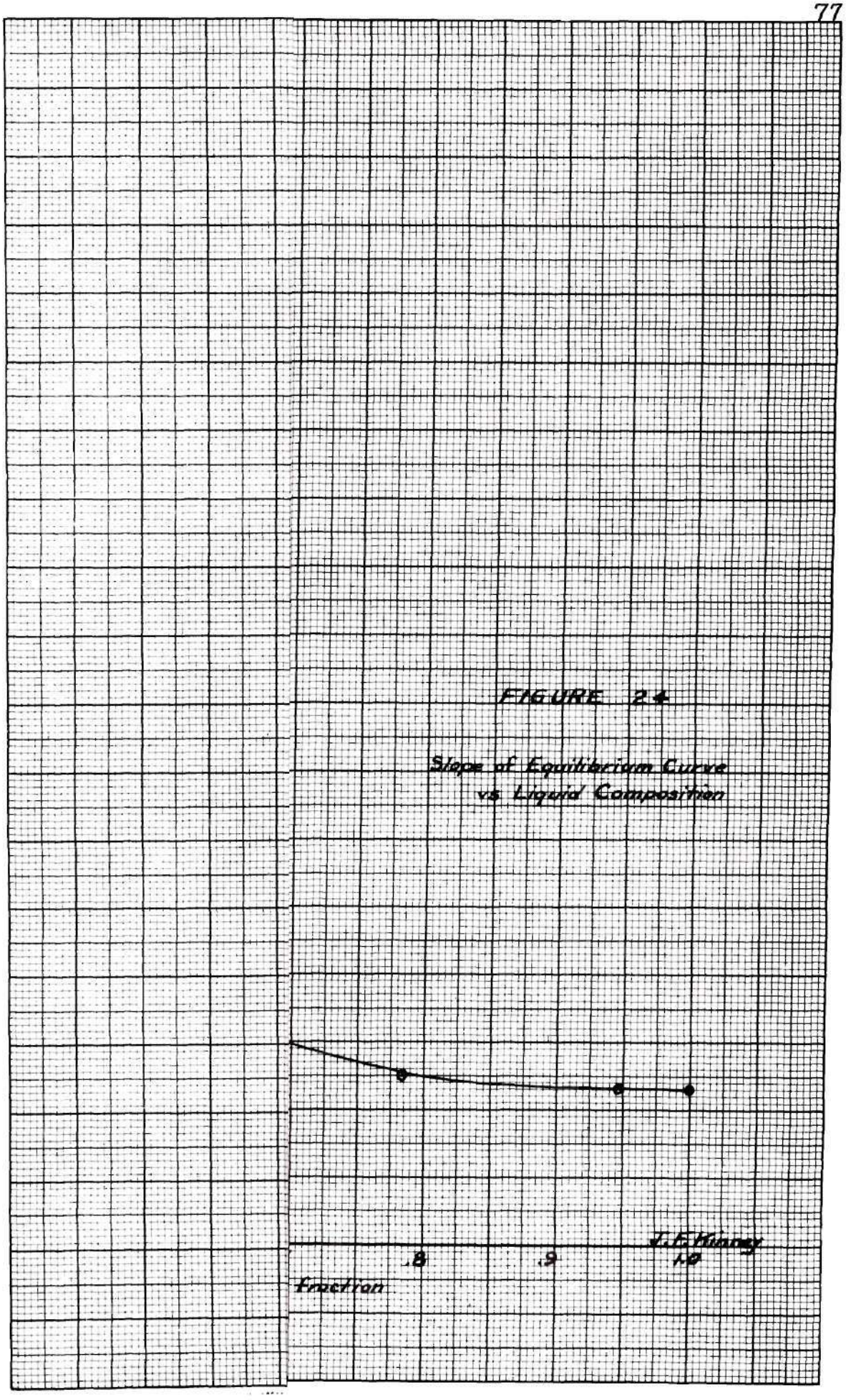


FIGURE 24
Slope of Equilibrium Curve
vs. Liquid Composition
.8
.9
1.0
Fraction
J. E. Kinney

APPENDIX III

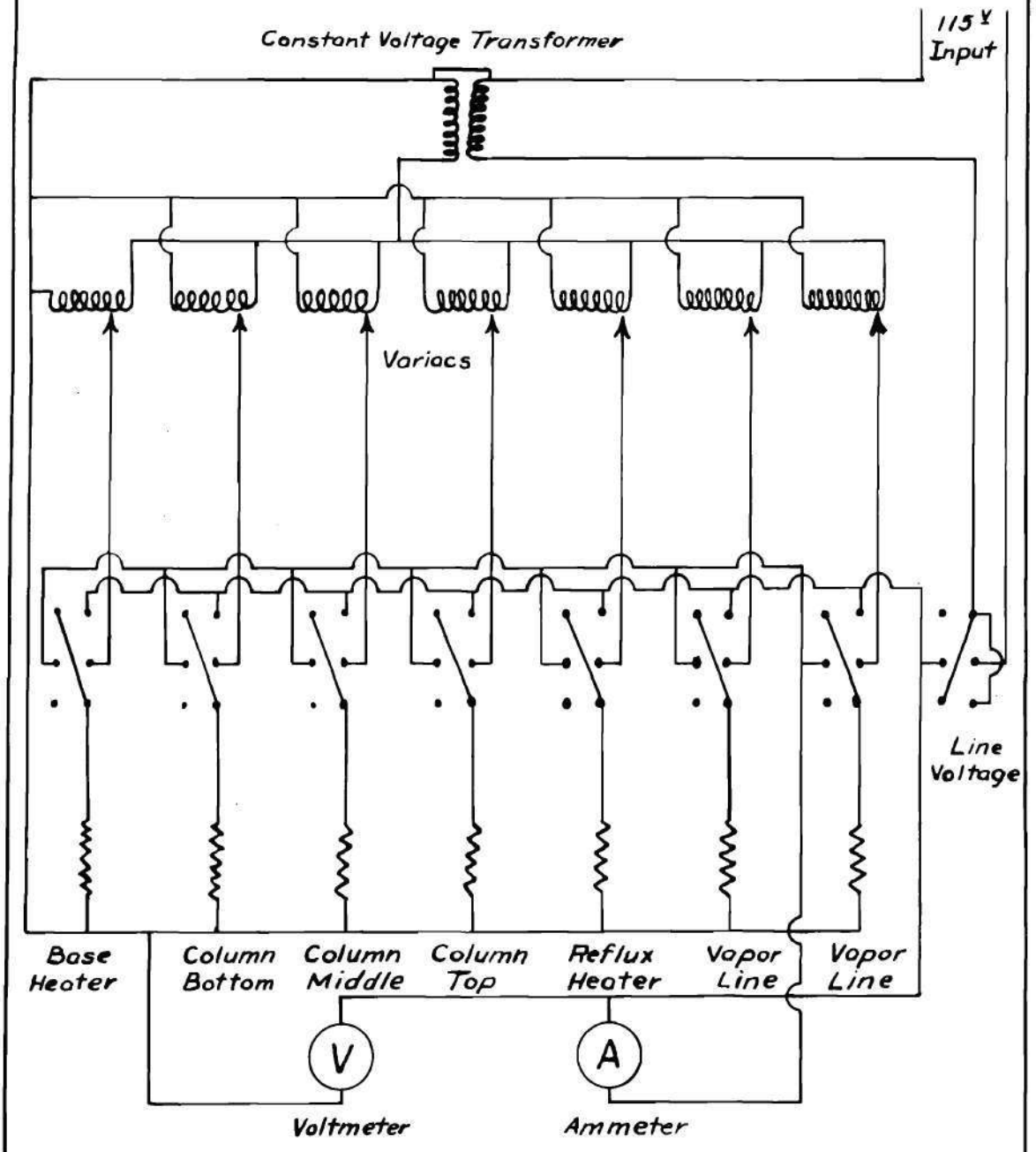


FIGURE 25

ELECTRICAL CIRCUIT

J.F. Kinney

APPENDIX IV

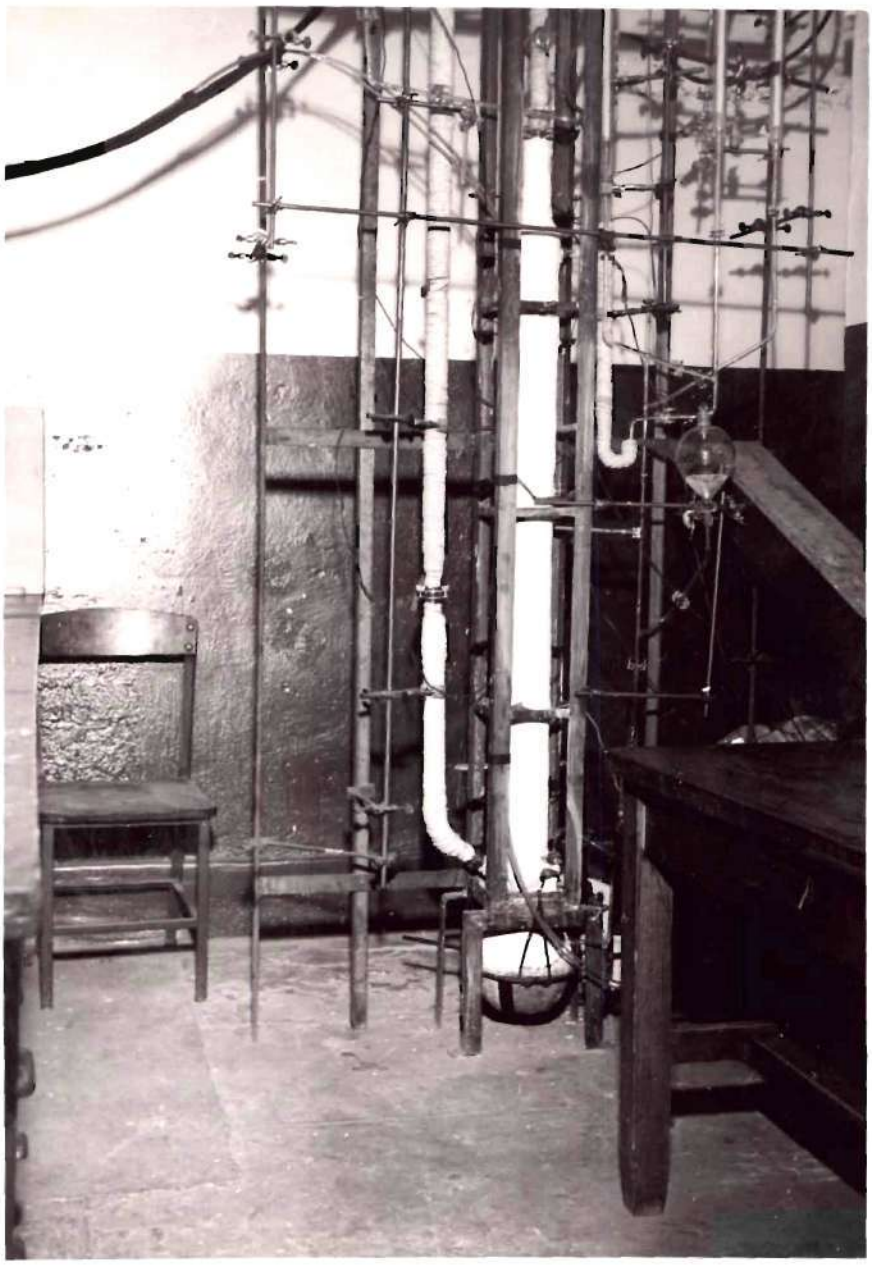


FIGURE 26

Lower Section of Apparatus with Column in Place

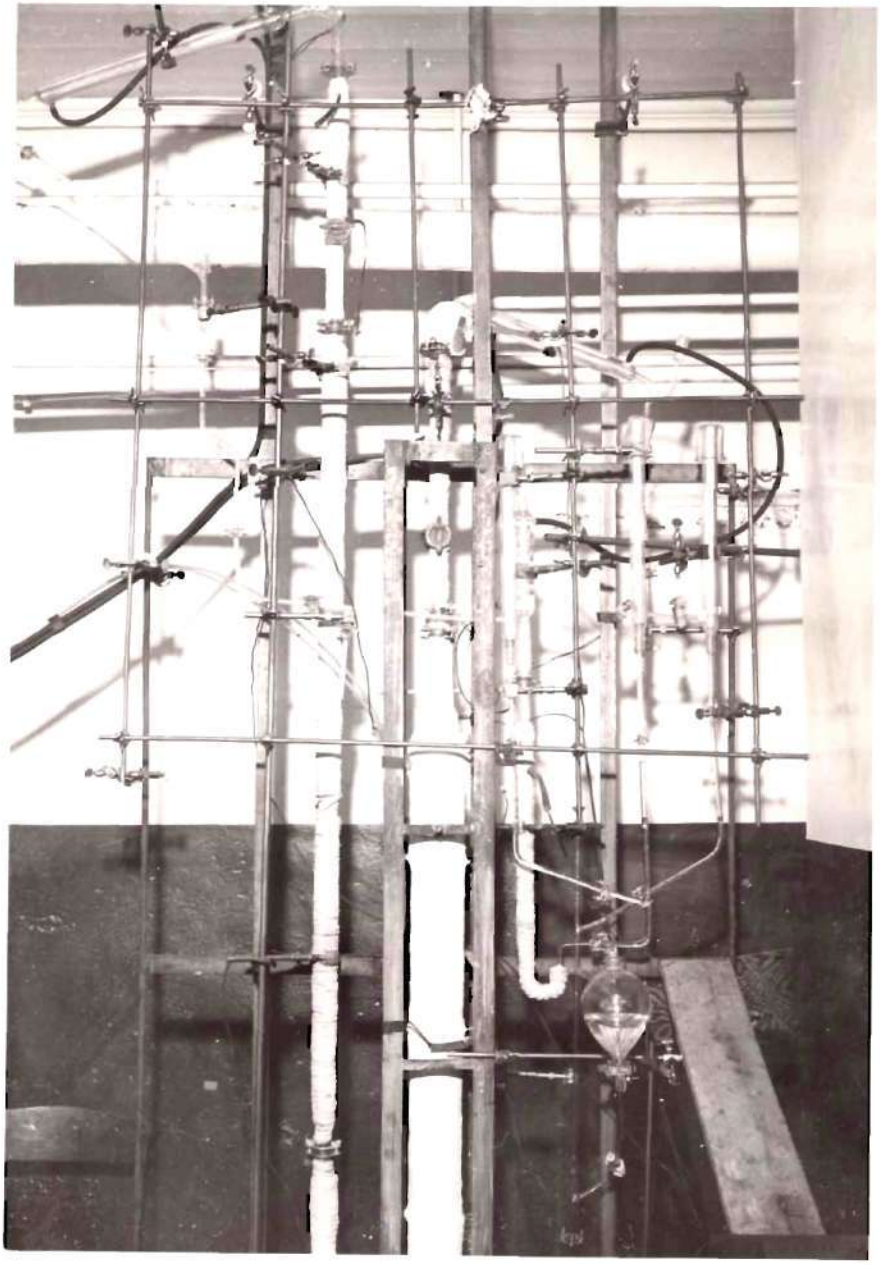


FIGURE 27

Upper Section of Apparatus Showing Arrangement of Flowmeters

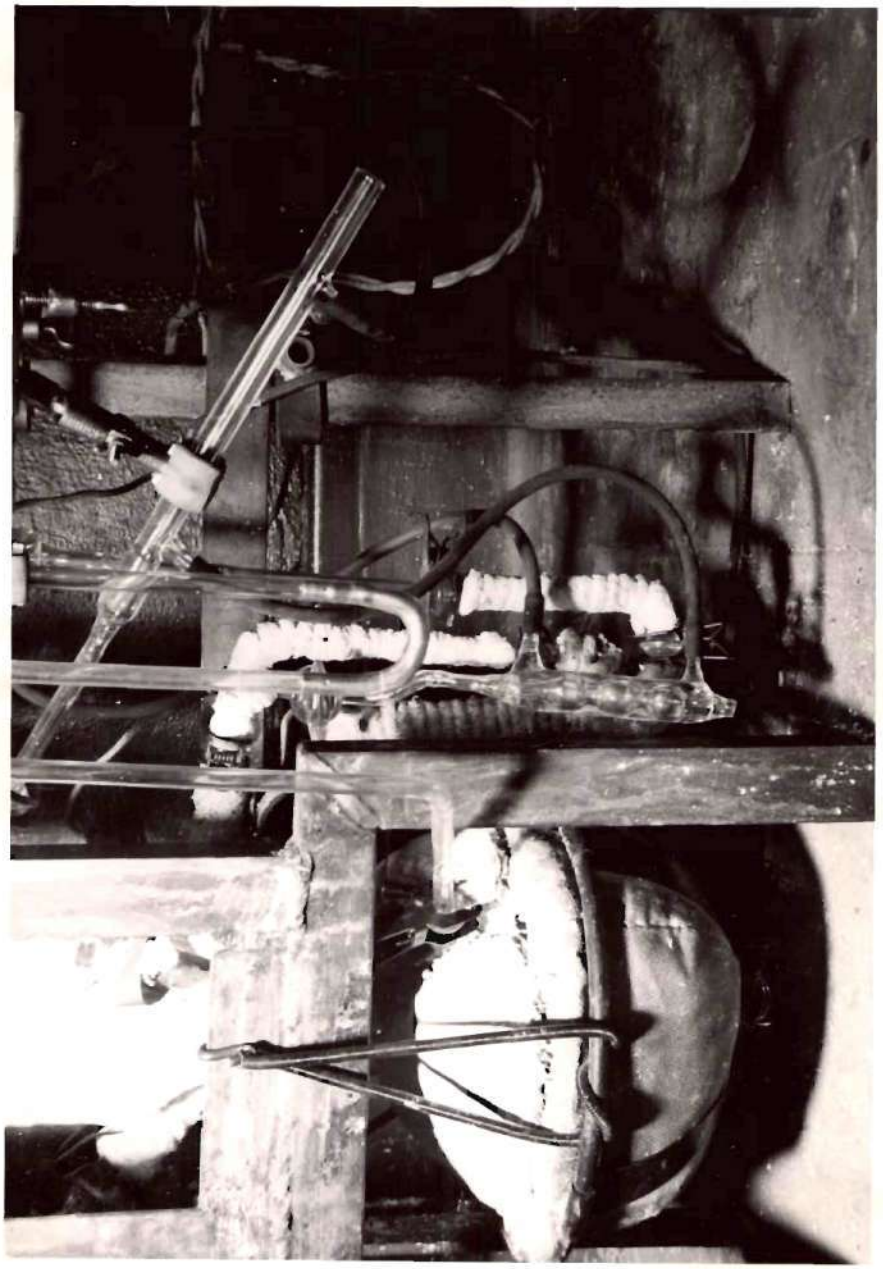


FIGURE 28

Close-Up of Boiling Flask Showing Vapor and Liquid Sampling Points

FIGURE 29

Control Panel